

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JANUARY 6, 1928.

I.—GENERAL; PLANT; MACHINERY.

Determination of the wettability of a solid by a liquid. Relation of adhesion tension to stability of colour varnish and lacquer systems. F. E. BARTELL and H. J. OSTERHOFF (Ind. Eng. Chem., 1927, 19, 1277—1280).—A method for determining the degree of wetting of a solid by a liquid is based on the measurement of the pressure of displacement of one liquid by another, *e.g.*, of water as it is driven from carbon black by organic liquids and of an organic liquid as it is driven from silica by water. For each system the adhesion tension, *i.e.*, the measure of the attraction of a solid for a liquid, has been calculated. This can be expressed in absolute units, and allows the relative capabilities of wetting of a solid by a series of liquids to be determined. Thus the relative stability of suspensions of the solid in the different liquids can be predicted. Other factors being equal, a suspension will be most stable in that liquid which shows the highest adhesion tension for the solid. When the suspension is present in a system of two immiscible liquids it will tend to go to that liquid which will give the higher adhesion tension with the solid; if in a mixture of two miscible liquids, the liquid which gives the higher adhesion tension with the solid will tend to displace the other liquid, and will finally give an absorbed layer round the solid. The application of adhesion tension determinations is discussed, and examples showing the relation of adhesion tension to the stability of suspensions in colour varnish and lacquer systems are given. E. H. SHARPLES.

Adhesives and adhesion: gums, resins, and waxes between polished metal surfaces. J. W. MCBAIN and W. B. LEE (J. Physical Chem., 1927, 31, 1674—1680; B., 1926, 291, 557).—Preliminary measurements show that joint strength increases with the thinness of the adhesive layer, especially when the thinnest possible films are considered. The effect of thickness becomes unimportant when the layer is more than 0.1 in. thick. The curves showing the effect of thickness of adhesive on joint strength in the cases of aluminium and nickel with a commercial shellac adhesive have a form similar to that obtained by Crow (B., 1924, 427) for lead-tin solder between copper surfaces. Data are given showing the adhesive power both in tension and in shear of various gums, waxes, and resins for the metals nickel, steel, cast iron, copper, brass, aluminium, tin, and lead. Waxes show failure mainly within the wax itself, and pitch is indicated as a cheap ingredient for use in adhesives. In general, the parallelism existing for joint strength between smooth metal surfaces and the mechanical and intrinsic properties of the metals

concerned is confirmed. Joint strength in shear is usually the same as in tension. L. S. THEOBALD.

Mill grinding. E. E. SPARROW (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1927, Circ. No. 319, 551—560).—Optimum conditions for stone mill grinding are discussed, attention being given to speed and setting of mill, style and method of dressing stones, etc.

S. S. WOOLF.

Strache-Kling explosion calorimeter, Löffler type. II. STRACHE and H. LÖFFLER (Gas- u. Wasserfach, 1927, 70, 1073—1077).—A glass, double-walled, explosion vessel is connected by a capillary to a pipette, the volume of which bears a known relation to that of the former, pipettes of several different sizes being supplied with the instrument. The gas to be examined is drawn, by means of a levelling tube, into the explosion vessel, air being drawn in after it to fill the latter. The mixture is exploded by a spark from an induction coil connected to the dry battery of a pocket lamp, and the temperature inside the explosion vessel is read before and after explosion by a thermometer so placed that its bulb hangs at the centre of the vessel. The rise in temperature, multiplied by a factor for the pipette used, gives the calorific value of the gas, which must be corrected for temperature and pressure. The calorimeter is generally applicable, and examples are given of determinations using hydrogen, carbon monoxide, methane, illuminating gas, and a mixture of 52.0% H₂, 37.8% CO, and 4.7% CH₄. Provided the correct volume of air to ensure complete combustion is mixed with the gas, the results are accurate to within 0.5%. The apparatus ("Caloriscopes"), mounted in a wooden case, is simple, self-contained, strong, and easily portable, and a determination can be made in 1½ min.

W. T. K. BRAUNHOLTZ.

Simplified refractometer for sugar and oils. F. LÖWE (Z. Ver. deut. Zucker-Ind., 1927, 690—695).—The latest Zeiss refractometer for sugar and oil work is described. Although similar in principle to the ordinary Abbé type, it is more compact in appearance, the scale and prisms being enclosed in a capsule-shaped casing fixed to the top of a standard on a very solid base. It has a double scale showing values for n_D (1.330—1.540) and corresponding weight-percentages of sugar solutions. The prisms have water jackets, and there is provision for taking readings by reflected light, *e.g.*, in the case of very dark liquids. J. H. LANE.

PATENTS.

Furnace. H. BANGERT (U.S.P. 1,649,648, 15.11.27. Appl., 9.6.26).—Below the top and extending from the charging towards the discharging end of a continuous

heating furnace is a wall, above which is a fuel-gas inlet adjacent to the charging end; extending longitudinally through the wall are passageways for supplying heated auxiliary air to the fuel gas adjacent to the other end of the furnace.

M. E. NOTTAGE.

Drying kiln. J. B. WELCH (U.S.P. 1,648,089, 8.11.27. Appl., 6.3.26).—Material to be dried is subjected to progressively decreasing humidity and increasing temperature as it travels through the kiln. Steam is passed through a short pipe at the dry end and thence through two pipes extending the full length of the kiln.

H. HOLMES.

Heat exchanger. J. E. KOBERNIK, ASSR. to NEWTON PROCESS MANUF. CO. (U.S.P. 1,648,646, 8.11.27. Appl., 22.6.26).—The main shell is provided with headers comprising three stationary chambers in one end and two movable chambers in the other end. Sets of tubes connect each of two stationary chambers to the corresponding movable chambers, and other tubes connect the movable chambers to the third stationary chamber. The shell space around the tubes is provided with an inlet and an outlet.

H. HOLMES.

Apparatus for rapidly heating to high temperatures small quantities of vaporisable solid material. CHEM. FABR. DR. H. STOLTZENBERG (G.P. 443,125, 14.11.25).—The flame from a lamp of the "Primus" type is directed inside a refractory fireclay cylinder enclosed in a perforated iron jacket and covered with an asbestos plate through a hole in the centre of which projects the lower half of a large quartz test-tube. Inside the latter is a second, somewhat longer, tube perforated at the lower end. Both tubes are closed with a common metal attachment provided with an annular ring in which the outer tube terminates and with a stopcock which closes the inner tube. In the movable portion of the stopcock is a slot for the reception of the material preferably in tabular form to be vaporised. Organic substances thus introduced may be rapidly vaporised at 700–1000° without carbonising; the vapours passing from the inner to the outer tube are decomposed, and the issuing gases may be suitably collected.

A. R. POWELL.

Drum for drying materials. J. CUYPERS (U.S.P. 1,648,542, 8.11.27. Appl., 16.7.26. Ger., 20.7.25).—A rotary drying drum is provided with carriers for the material extending parallel to its axis. Each carrier comprises three radial arms 120° apart with flanges extending from them in the direction of rotation.

H. HOLMES.

Drum dryer with built-in cells. M. KIRCH (G.P. 442,545, 17.6.24).—The pyramidal or truncated-conical draining surfaces of the scoops are inclined to the drum axis at an angle which is either equal to or greater than the angle of repose of the material to be dried. The cellular screens are arranged either in close succession or in groups.

J. S. G. THOMAS.

Pulveriser. C. M. STONER, ASSR. to WOLF CO. (U.S.P. 1,648,747, 8.11.27. Appl., 30.1.26).—A crusher roll consists of a shaft to which is secured a series of driving discs with spindles extending between them, and another driving disc provided with a series of spindles which, in turn, carry a tension disc.

M. E. NOTTAGE.

Pulverising apparatus. L. V. ANDREWS, ASSR. to RILEY STOKER CORP. (U.S.P. 1,646,720, 25.10.27. Appl., 18.5.26).—A pulveriser comprising revolving beaters is provided with means, such as smaller beaters rotating within a perforated conical sleeve, to deflect hard particles out of the main pulverising chamber.

B. M. VENABLES.

Pulveriser. H. A. KIMBER (U.S.P. 1,646,752, 25.10.27. Appl., 20.8.26).—In a pulveriser comprising a substantially circular chamber containing several sets of beaters, a set or sets of stationary baffles are provided between the sets of beaters shaped so as to cause material flung by one set of beaters to rebound into the path of another set.

B. M. VENABLES.

Rotary pulveriser. W. H. HARTMAN, ASSR. to BONNOT CO. (U.S.P. 1,647,730, 1.11.27. Appl., 12.1.27).—Rotating hammers are mounted in a cylinder provided with an inlet for the material, and a grinding surface within the cylinder is provided with passages for discharge of ground material. A breaker block within the cylinder deflects heavy particles against a door communicating with a trap external to the cylinder and adapted to open only when such particles are thrown against it.

H. HOLMES.

Disintegrating mill. A. LEGGEMANN (U.S.P. 1,647,183, 1.11.27. Appl., 26.2.27. Ger., 31.1.27).—Rotary beaters pass between bars of a grate forming the walls of a hopper for the materials. The ends projecting into the hopper co-operate with a rigid abutment located above its lower end and spaced from the lower ends of the bars.

H. HOLMES.

Dry-pan grinding apparatus. W. L. HANLEY, JUN. (U.S.P. 1,648,176, 8.11.27. Appl., 14.10.24).—A stationary rim formed of separate sections having overlapping parts is arranged slightly above a horizontal rotatable pan and forms a discharge aperture between its lower edge and the pan. Each section has outwardly extending brackets rigidly secured to it, and devices provided with screw-threaded adjusting means for engaging these brackets enable the rim sections to be adjusted independently and retained against accidental movement.

J. S. G. THOMAS.

Crusher and pulveriser. H. J. SHELTON (U.S.P. 1,648,625, 8.11.27. Appl., 11.2.27).—A housing encloses a pulverising chamber provided with rotary hammers and a pocket opening into the chamber at a point above the path of the hammer tips. The pocket collects foreign objects from the material in the chamber, and its wall, contiguous with the upper wall of the chamber, has passages for the return to the chamber of any reduced material collected.

H. HOLMES.

Dewatering apparatus for pulp. J. STEPHANSEN (U.S.P. 1,650,100, 22.11.27. Appl., 4.5.26. Norw., 4.4.25).—A rotatable perforated drum forms one side wall of a pulp container, and an endless felt or sieve passes partly through the container and partly around the drum. Means are provided for preventing passage of fluid through the perforations at that part of the periphery of the drum which is not in contact with the felt.

J. S. G. THOMAS.

Continuous filter. A. C. DAMAN (U.S.P. 1,650,434, 22.11.27. Appl., 11.7.25).—In the separation of solution

from pulp, the latter, contained in a tank, is conveyed to the inside of a drum having an internal, cylindrical, filtering surface and rotating, partly submerged, in the tank.

H. ROYAL-DAWSON.

Séparator. L. E. BROWN (U.S.P. 1,648,607, 8.11.27. Appl., 13.11.26).—A conical separating chamber is provided with a supply connexion, and with a series of superposed rotary members arranged beneath the normal level of the liquid. Means are provided for withdrawing clarified liquid from beneath each member at a point near the centre of the chamber and for removing foreign matter accumulating on the surface of the liquid as well as that precipitated.

H. HOLMES.

Rotary screening apparatus. J. WATT and T. A. POOL (E.P. 278,939, 12.2.27).—A form of apparatus where a number (usually four) of cylindrical screens are grouped and rotate round a common shaft. Provision may be made for a longitudinal bumping movement to be also applied to the screens.

B. M. VENABLES.

Rotary separators. PNEUMATIC CONVEYANCE & EXTRACTION, LTD., and W. A. SMITH (E.P. 278,900, 8.11.26).—An apparatus for separating material that is being pneumatically conveyed comprises a receiving hopper in which the large material drops directly out and above which rotate one or more discs provided with upstanding angle-irons or vanes which remove the remainder of the solid matter by centrifugal action.

B. M. VENABLES.

Mixing apparatus. N. C. JOHNSON (U.S.P. 1,646,540, 25.10.27. Appl., 31.3.26).—A batch of ingredients is mixed with compacting and attrition between the particles by subjecting it, while confined by the wall of a container, to centrifugal force such as will produce a flow of the mass at high velocity. A portion of the batch is intercepted and retarded to form a wedge. This diverts the mass while still compacting it, and causes it to flow into position for further compacting under the centrifugal action. The cycle is repeated until the batch is thoroughly mixed.

H. HOLMES.

Mixing device. R. BOWEN, Assr. to SUPER COAL PROCESS Co. (U.S.P. 1,646,386, 25.10.27. Appl., 24.2.25).—A mixer for heterogeneous material which may be viscous comprises upper and lower hoppers and a mixing chamber between, the latter having a tiltable piece in the bottom forming a large opening into the lower hopper, and both hoppers having valve-controlled outlets. The mixing device comprises blades which produce both vertical and horizontal movement in the material.

B. M. VENABLES.

Manufacture of filtering material. H. BLUMENBERG, JUN. (U.S.P. 1,649,386, 15.11.27. Appl., 14.4.27).—The material consists of a fluffy porous cellular material intimately mixed with lead oxide.

H. ROYAL-DAWSON.

Production of anti-freezing solutions. G. A. SCHULTHEISS (U.S.P. 1,649,261, 15.11.27. Appl., 10.1.27).—Crushed or ground vegetable matter containing fats or oils is treated with calcium chloride solution.

H. ROYAL-DAWSON.

Hardness-testing machine. H. M. GERMAN (U.S.P. 1,646,195, 18.10.27. Appl., 29.5.26).—A member is brought into engagement with the material to be tested and a predetermined relative movement between

them is effected gradually under a predetermined constant pressure exerted by a weight. The member is thus caused to penetrate the material, to an extent determined by the hardness, whereupon the member is returned to its original position.

H. HOLMES.

Apparatus for determination of viscosity of fluids. W. J. ALBERSHEIM and H. S. KONHEIM (E.P. 263,781, 8.12.26. U.S., 23.12.25).—The liquid (e.g., lubricating oil) is pumped through a turbine or other engine which drives a braked governor, whereby its speed is held constant and therefore the flow of oil is constant. The oil continues through a narrow-bore tube or constriction to a tank not under pressure, and the pressure between the turbine and the constriction is registered, this pressure being a measure of the viscosity of the oil.

B. M. VENABLES.

Colour-testing device for translucent fluids. E. KADDATZ (U.S.P. 1,647,569, 1.11.27. Appl., 28.1.26).—A column of predetermined thickness of the sample under test in a transparent container is mounted against a sharply contrasted coloured background and compared with a band bearing colours corresponding to varying grades of opacity of the liquid.

H. HOLMES.

Indicating or controlling devices operating in the presence of impurities or other additions to gases or liquids. DEUTS. GASGLÜHLICHT-AUER-GES. M.B.H., K. WOLLIN, and E. SMOLCZYK (E.P. 278,927, 17.1.27. Addn. to E.P. 254,299; B., 1927, 832).—The destructible pellet of the original patent is supported on fingers of fusible metal, by which means degrees of heat will also be indicated.

B. M. VENABLES.

Filter. T. F. SEITZ and G. H. SEITZ, JUN., Assrs. to SEITZ-WERKE G.M.B.H. (U.S.P. 1,650,543, 22.11.27. Appl., 18.3.26. Ger., 25.3.25).—See B.P. 249,857; B., 1926, 807.

Centrifugal separating apparatus. F. GRIMBLE, M. N. CAIRD, and E. COOMBS (U.S.P. 1,649,346, 15.11.27. Appl., 18.11.26. U.K., 20.10.25).—See B.P. 269,218; B., 1927, 433.

Absorption refrigerating apparatus. ELECTROLUX, LTD., Assees. of B. C. VON PLATEN and C. G. MUNTERS (B.P. 259,240, 2.10.26. U.S., 2.10.25.).

Apparatus [container] for use in the purification of substances by means of radio-active materials. M. DICKINSON (B.P. 280,324, 15.9.26).

Chemical fire extinguisher. S. F. BARCLAY and MATHER & PLATT, LTD. (B.P. 280,388, 13.12.26).

Production of fire-extinguishing foam. EXCELSIOR FEUERLÖSCHGERÄTE A.-G. and H. BURMEISTER, Assees. of MINIMAX A.-G. (B.P. 256,245, 28.7.26. Ger., 30.7.25).

II.—FUEL; GAS; TAR; MINERAL OILS.

Microstructure of New Zealand lignites. W. P. EVANS (New Zealand J. Sci. Tech., 1927, 9, 137—158; cf. B., 1927, 690).—Etched surfaces and thin sections of Steventon lignite have been examined microscopically, both in the unaltered state and as modified by a dolerite intrusion. To the eye the coal sometimes appears uniform, sometimes definitely banded, the smaller bands often consisting of a single flattened stemlet. The

microstructure shows it to have been formed chiefly from coniferous woods. Cuticle is found frequently, and spores occur definitely though comparatively rarely. They are mostly megaspores of comparatively small dimensions (120—410 μ in length). The dolerite intrusion has obliterated much of the observable organic structure, of which, however, traces can still be recognised. The resin constituents, though in small amount, are more evident in the altered than in the unaltered lignite. The secondary mineral matter in the unaltered lignite is fairly evenly distributed; silica occurring as cryptocrystalline chalcedony and in unbanded colloidal masses accounts for much of the ash. The even distribution is disturbed in the altered lignite, which is finely fissured, the fissures containing mineral matter derived from the dolerite or from the subsequent infiltration of clays. Sulphur has been transferred from the organic to the inorganic compounds by the action of the dolerite intrusion. The coal-forming deposit consisted chiefly of transported material, and was laid down under water.

A. B. MANNING.

Lignites of Ribolla. M. G. LEVI and C. PADOVANI [with A. AMATI, F. BURRAL, C. SINIRAMED, G. GRIFFI, and E. SALMOIRAGHI] (*Annali Chim. Appl.*, 1927, 17, 491—512).—When lumpy and fine residual lignite from the pits at Ribolla, in the province of Grosseto, were distilled at 500° in a laboratory Fischer rotating furnace, they yielded 76.5% of semi-coke, 7.5% of tar, 5.4% of combined water, and 62 litres of gas (giving 6800 kg.-cal. per cub. m.) per kg. The lignite contains much nitrogen but only little sulphur, and has a calorific value exceeding 5500 kg.-cal. on the dry material. The semi-coke obtained is non-agglomerating and retains the form of the original lignite, its ash being 30% and its calorific value 5400 kg.-cal.; it retains about 90% of the total nitrogen. The tar contains 2—3% of paraffin, small proportions of phenols, 17% of light products (b.p. to 200°), and 40% of oils of b.p. 200—300°. When submitted to hydrogenation by the Bergius process, the lignite, the residue left when it is semi-distilled at 250°, the semi-coke, and the tar give poor yields of oil.

T. H. POPE.

Behaviour and economics of gas-works and coke-oven cokes when used for firing a central-heating boiler. W. LEDER (*Gas- u. Wasserfach*, 1927, 70, 1045—1047).—A comparison of the mean figures of a large number of analyses of German gas-works and coke-oven cokes used for central-heating boilers shows no appreciable difference between them in moisture or ash content or in calorific value. A practical test in a central-heating boiler, using a gas-works and coke-oven coke of about the same analysis, also showed no difference in their heating efficiency. The price is thus the deciding factor in estimating their relative value.

W. T. K. BRAUNHOLTZ.

Determination of melting points of ash in coal. D. J. W. KREULEN (*Chem. Weekblad*, 1927, 24, 599—602).—The general rule that the m.p. of the ash becomes higher as the ash content increases, and therefore with the fineness of the coals, is confirmed, but exceptions are noted. The use of dextrin for the preparation of the cone is not recommended. Addition of pulverised coke

from the coal itself may raise the softening point of the ash cone by as much as 175°. For standard determinations, the cone should be prepared by mixing the finely-powdered ash with distilled water and pressing in a mould; very uniform temperatures are found in this way. If the m.p. is above 1250°, no difficulty should be found with steady firing; this is regarded as the critical point.

S. I. LEVY.

Deflocculation of carbon black by saponin, acacia, gelatin, and casein. R. M. CHAPIN (*Ind. Eng. Chem.*, 1927, 19, 1275—1277).—An extension of previous work (cf. B., 1927, 117). The carbon black test has been used to study the deflocculating powers of commercial samples of saponin, acacia, gelatin, and casein compared with that of potassium palmitate. Both "naked" and "oiled" black have been used, and the effects of variation in the conditions and of the presence of octyl alcohol have been examined. Potassium palmitate and saponin deflocculated "naked" black more powerfully than they did the "oiled" black. Casein in alkaline solution, and probably also in acid solution, acacia in the presence of octyl alcohol, and especially gelatin deflocculated "oiled" black more powerfully than they did the "naked" black. Against "naked" black saponin seemed more powerful in neutral solution, and even the addition of sodium chloride was detrimental, whilst the powers of acacia and particularly gelatin were enhanced by the presence of acid. Octyl alcohol appeared slightly beneficial to soap and strongly so to acacia, whereas in all other cases it was distinctly detrimental, especially to gelatin.

E. H. SHARPLES.

Colour standards for bone-black revivification tests. R. BAUS (*Ind. Eng. Chem.*, 1927, 19, 1296).—For judging the efficiency of carbonisation in the kiln, 60 g. (measured) of the revivified char are boiled for 2 min. with 75 c.c. of caustic soda solution (*d* 1.036) in a 250 c.c. covered copper beaker and filtered. The colour of the filtrate is compared with a series of standards prepared by adding to 35 c.c. portions of 0.5% acetic acid, 0.25, 0.50, 1.0, and 2.0 c.c. of a 0.04% solution of Bromothymol Blue. The standards, representing "over-burnt," "well-burnt," "mediocre," and "poor" chars, respectively, are stable for several weeks if tightly corked and sealed with paraffin.

E. H. SHARPLES.

Reaction products obtained by the decomposition of paraffin at 450° ("cracking") and by its decomposition in the presence of hydrogen at high pressures ("berginisation"). H. I. WATERMAN and J. N. J. PERQUIN (*Rec. trav. chim.*, 1927, 46, 813—836; cf. B., 1926, 307).—The products obtained by application of the two processes under almost identical conditions show great similarity, fractions of practically the same b.p. being obtained in each case. The cracking products are characterised in contrast to berginisation products by their higher bromine numbers and refractive dispersions and by their lower aniline indices and stability. It is suggested that cracking takes place first in berginisation, and is followed by the hydrogenation of the unsaturated hydrocarbons. The hexane, heptane, and octane fractions from cracking contain about twice as much olefine as the corresponding fractions from berginisation. In cracking there are indications of

fractions with b.p. between those of the normal hydrocarbons which have a high refractive dispersion and low unsaturation and aniline point values. They are possibly of an aromatic character. The refractive dispersions and the bromine values of mixture of *n*-hexane and Δ^8 -hexene follow a straight-line law.

G. A. C. GOUGH.

Composition of water-gas at low temperatures. W. REINDERS (Z. physikal. Chem., 1927, 130, 405—414). The composition of water-gas from 200° to 1000° has been calculated from the constants of the equilibria $\text{CO}_2 + \text{H}_2 \rightleftharpoons \text{CO} + \text{H}_2\text{O}$, $2\text{CO} \rightleftharpoons \text{C} + \text{CO}_2$, and $\text{C} + 2\text{H}_2 \rightleftharpoons \text{CH}_4$, by means of the equation $p_{\text{CO}} + p_{\text{CO}_2} + p_{\text{H}_2} + p_{\text{CH}_4} + p_{\text{H}_2\text{O}} = 1$. At temperatures below 400°, the ideal water-gas consists of a mixture of equal parts of carbon dioxide and methane, together with water vapour. It is shown how the formation of methane in nature can be explained by a reaction between carbon and water. The possibility of the formation of higher hydrocarbons at low temperatures is discussed.

L. L. BIRCUMSHAW.

Fuel for motor transport. IV. Power alcohol from grasses, straws, and waste vegetable materials (Dept. Sci. Ind. Res., Fuel Res., 1927, 26 pp.).—From a study of the hydrolysis of wheat straw and other waste vegetable materials to sugars, and the fermentation of the latter to give power alcohol, the conditions were determined which were most satisfactory for technical application of the process. The straw etc. was soaked for 4 hrs. in a 2.0—2.5% sulphuric acid solution, and the damp material, from which excess acid had been removed by draining and then washing with cold water, was steamed for 6 hrs. This converted the hemicelluloses completely into pentoses, which were extracted with sterile water. After adding lime to neutralise the acid, the extract was fermented by *B. acetohylicus*, thereby yielding about 33% (calculated on the pentoses) of an alcohol-acetone mixture containing about 7% of acetone. The steaming process sufficed for the destruction of the normal microflora of the raw material, so dispensing with the necessity for a separate sterilisation of the mash. The yields of alcohol-acetone mixture varied from 10 to 22.5 gals. per ton when the process was carried out on a semi-technical scale, using raw materials such as tropical and semi-tropical grasses, straws, rice husks, etc. from different parts of the Empire. Estimates are given of the costs of production. The present position of the use of suction-gas producers on motor lorries is briefly summarised.

A. B. MANNING.

Shale oil. I. Genesis of oil shale in relation to petroleum and other fuels. II. Gases from oil shale. R. H. MCKEE and P. D. V. MANNING (Oil Bull., 1927, 13, 65—69, 175—181, 291—301, 489—493, 729—737, 837—843).—The chemical and geological evidence is reviewed, and the origin of kerogen and oil is discussed. A modified gas-analysis apparatus is described; 85% phosphoric acid is employed in the determination of ammonia. Kerogen is regarded as losing carbon dioxide from its carboxyl groups before soluble bitumen or oil is formed; it contains no hydrocarbons as such. Nitrogen is evolved as ammonia in two stages. Kerogen is placed in the series of carbonaceous substances from which coal is formed; on pyro-

lysis it yields bitumen, soluble bitumen, and oil, with evolution of gas accompanying each stage. Kerogen is believed to be essentially the resinous residues from a past vegetation.

CHEMICAL ABSTRACTS.

Action of antidetonators on the adiabatic inflammability of hydrocarbons. M. AUBERT, A. PIGNOT, and J. VILLEY (Compt. rend., 1927, 185, 1111—1113).—The addition of lead tetraethyl in increasing amounts (up to 10%) to cyclohexane raises the characteristic adiabatic inflammability curve slightly, to an extent which depends roughly on the concentration, and is almost negligible when this is small. *n*-Heptane and *n*-hexane behave similarly, but large concentrations produce a slight reduction in the inflammability value. The results, which are analogous to those previously obtained (B., 1926, 260), indicate an absence of parallelism between the effects of "anti-knock" compounds on the detonation limit and on the adiabatic inflammability, and support the theory that liquid droplets play an essential part in the determination of anti-knock effects.

J. GRANT.

Gas calorimeter. STRACHE and LÖFFLER.—See I. **Determination of constituents of gaseous mixtures.** WILMET.—See VII. **Sucrose and adsorbent carbons.** VAŠÁTKO.—See XVII. **Safety explosives.** NAOUM.—See XXII.

PATENTS.

Vertical retort. J. TRAUTMANN (G.P. 442,608, 13.4.26. Addn. to G.P. 430,365; B., 1927, 468).—One or more concentric rings or segments are placed between the conical heating and sliding surfaces in the apparatus described previously, to prevent congestion of the space between the surfaces by the material under treatment.

L. A. COLES.

Distillation retort. F. RICHTER (G.P. 442,772, 27.6.23).—Helical flues are built in the walls of a vertical retort, ample wall space being left between each coil, and the cross-section of the flues being such that they are easily cleaned.

L. A. COLES.

Retort for low-temperature carbonisation. COMP. DES MINES DE BRUAY (F.P. 618,464, 3.7.26).—An inclined rotating retort contains metal rollers to facilitate conduction of heat and to prevent caking of the charge.

L. A. COLES.

Oil-gas producer. O. MISCH (G.P. 442,421, 21.7.25).—Oil or tar is distilled in an externally heated retort filled with coke which, charged with oil or tar residues, is discharged at the lower end of the apparatus and is burnt to supply the necessary heat, or is used for the production of gas, part of which is burnt and the remainder is used for flushing out the retort.

L. A. COLES.

Water-gas producer. M. H. HERENG (F.P. 618,552, 12.11.25).—A moving grate conveys glowing coal through a tunnel over apparatus which supplies the necessary air and superheated steam.

L. A. COLES.

Treatment of fuel. COMP. DES MINES DE BRUAY (F.P. 617,522, 14.6.26).—Agglomerated or briquetted fuel encased in granular material, such as sand or kieselguhr, is subjected to low-temperature carbonisation to produce semi-coke, which is used as smokeless fuel.

L. A. COLES.

Improvement of fuel. F. GROLEAS and D. ARNAUTOVITCH-D'ALBANY (F.P. 615,967, 3.10.25).—The fuel is sprayed with a mixture of an alkali nitrate and acetic acid, which may also contain sodium chloride and citric acid. L. A. COLES.

Manufacture of gas. WOODALL-DUCKHAM (1920), LTD., E. W. SMITH, and T. C. FINLAYSON (B.P. 278,486, 25.8.26).—In a system of continuous vertical retorts in which one or more retorts are used for the manufacture of water-gas (cf. B.P. 253,702; B., 1926, 699) the liquor in the common collecting main is divided in compartments each corresponding with a separate retort. There are also two independent liquor circulation systems to each compartment according to the gas made. Where there are two collecting mains common to all the retorts the washing liquor is supplied separately to each main. A. C. MONKHOUSE.

Recovery of sodium thiocyanate [from gas liquors]. R. E. HALL, Assr. to KOPPERS Co. (U.S.P. 1,648,224, 8.11.27. Appl., 4.4.22).—The mother-liquors from gas purification are evaporated to dryness, the residue being then treated with a selective solvent, *e.g.* alcohol, which extracts the thiocyanate without dissolving alkali sulphates and carbonates. The solution after passing through a charcoal filter is evaporated. H. ROYAL-DAWSON.

Steam distillation of coal etc. SYNTHETIC AMMONIA & NITRATES LTD., and R. E. SLADE (B.P. 278,577, 21.2.27).—The fuel is distilled in steam and the products of distillation are cooled to a temperature above the dew-point by spraying with water or oil in a packed scrubbing tower (cf. B.P. 276,522; B., 1927, 868). Tar and oils are deposited in a dry condition and can, if required, be fractionally condensed. The dirty steam is used for the generation of steam in a boiler or heat-exchanger, which steam after compression and superheating is used for further distillations. A. C. MONKHOUSE.

Calcining of coke [for electrodes]. ALUMINUM CO. OF AMERICA, Assees. of V. C. DOERSCHUK and F. C. FRARY (B.P. 269,849, 6.1.27. U.S., 22.4.26).—Coke such as the by-product from the refining of crude petroleum is fed into a continuous vertical retort of oval or rectangular cross-section which is heated electrically at a point half way down the retort by electrodes arranged on the major axis at right angles to the flow of coke. The coke is cooled by passing up from the bottom of the retort a portion of the carbonisation gases which have been washed and cooled. A. C. MONKHOUSE.

Distillation of fuel by a current of hot gases. METALLBANK & METALLURGISCHE GES. A.-G. (G.P. 442,838, 2.12.25).—The use of two or more superposed distillation zones arranged parallel to the apparatus for charging the gas and fuel into the retort renders possible the economical distillation of small fuel. L. A. COLES.

Apparatus for carbonisation of wood. M. DAVÈNE (F.P. 617,814, 14.10.25).—The apparatus consists of a container fitted with partition walls arranged so as to form three concentric retorts with double walls between which hot gases circulate. L. A. COLES.

Manufacture of active carbon. SOC. POUR L'EXPLOIT. DES PROC. E. URBAIN (B.P. 255,871, 21.7.26. Fr.,

25.7.25).—In order to pulverise active carbon without loss of activity the material is crushed in a neutral liquid having a high surface tension, *e.g.*, water, oils, or hydrocarbons. The carbon can be subsequently washed and dried under diminished pressure. A. C. MONKHOUSE.

Apparatus for crushing and mixing pitch. PRÉPARATION IND. DES COMBUSTIBLES (B.P. 271,903, 28.5.27. Fr., 29.5.26).—The pitch passes through a single-cylinder crusher to a Carr grinder which has scrapers to remove the pitch into the foot of an elevator. This elevator is connected to the suction side of the grinder, which causes a current of air to be drawn through the grinder. From the elevator the pitch is discharged into the mixer, which consists of a rotating plate with openings closed at the bottom by pistons in order to discharge the pitch. The height of the piston controls the amount of material discharged, which is removed by a scraper in a continuous stream. A. C. MONKHOUSE.

Manufacture of gasoline. F. E. GILMORE, Assr. to F. AHLBURG and W. K. WHITE (U.S.P. 1,649,345, 15.11.27. Appl., 5.8.24).—Vapours containing recoverable gasoline are led into the bottom of a vertical chamber and rise through a series of baffles to the outlet pipe at the top. The vapours are drawn off through a check valve by a compressor, condensed under pressure, and collected in an accumulator tank. Residual gas leaves the system at the top of this tank through a discharge pipe fitted with a valve controlled by the temperature of the chamber. The condensate is returned to the top of the chamber and flows downwards over the baffles in counter-current to the ascending vapours. The recovered gasoline collects at the bottom of the chamber. R. C. ODAMS.

Recovery of gasoline. E. C. HERTHEL and T. DE C. TIFFT, Assrs. to SINCLAIR OIL & GAS CO. (U.S.P. 1,648,585, 8.11.27. Appl., 5.7.24).—Gasoline, absorbed in a liquid medium, is separated therefrom by distillation under pressure, the heated liquid passing to vertical columns wherein the pressure is reduced by virtue of the hydrostatic head of the liquid in the column, and the residual liquid passing in counter-current flow to (and in direct contact with) the resulting vapours. C. O. HARVEY.

Treatment of lubricating oils. ALFA-LAVAL CO., LTD. From N. E. FUNK (B.P. 279,636, 29.10.26).—Acid oxidation products are removed from used lubricating oil by washing the oil with the necessary quantity of water in a U-shaped circulating tank. Air is excluded during the process. The washed oil is freed from water, acid extract, and sludge by treatment in a centrifuge and subsequent filtration through a colloidal filter. The process may be continuous or intermittent, and the apparatus may be directly connected to the oil reservoir in the oil-circulating system of a turbine or other machine. R. C. ODAMS.

Treatment of acid sludge or acid resin obtained from the refining of mineral oils. J. JUBB. From J. PICKERING (B.P. 279,613, 4.10.26).—Acid sludge is dissolved in an organic solvent (*e.g.*, acetone) miscible with water or aqueous solutions, at a temperature at least 5° below the b.p. of the solvent. The resulting solution is treated with water, with continual stirring, and the temperature raised to a point slightly above

the b.p. of the solvent, the amount of water to be added being dependent on the acid content of the sludge and the required concentration of the aqueous extract. The solvent is driven off, and passes to a condenser, where it is recovered; the aqueous acid solution is drawn off from the bottom of the containing vessel and concentrated, and the residual sludge is heated at a slightly higher temperature (5–15° above the b.p. of the solvent), treated with a dilute solution of sodium chloride or carbonate with continuous agitation, and the temperature further raised to about 80°, more of the organic solvent being thus removed. The washing liquid is drawn off and the neutral sludge freed from last traces of solvent by heating it at 125°. The recovered solvent is used for dissolving a fresh charge of acid sludge. The solvent is not recovered from the wash liquid, but the latter is used again until the concentration of dissolved solvent is sufficiently high, when the liquid may be concentrated or used for directly treating acid sludge. R. C. ODAMS.

Distillation of hydrocarbon oils. F. A. HOWARD, ASSR. to STANDARD DEVELOPMENT Co. (U.S.P. 1,649,532, 15.11.27. Appl., 12.8.20).—Pyrogenetic distillation of oil is effected by passing a stream of the oil upwards through a heating zone into a main supply maintained under pressure. Steam (3–7%) is injected into the oil at a point between the heating zone and the main supply. R. C. ODAMS.

Destructive distillation of hydrocarbons. C. W. TURNER (U.S.P. 1,647,026, 25.10.27. Appl., 25.7.23).—A heated mixture of oil and steam, formed in a large vertical chamber, is caused to flow in a restricted stream to the bottom of another similar chamber, whereby expansion occurs and the mixture passes to the top of the second chamber and thence successively to two more similar chambers heated at a high temperature (about 1094°). The evolved vapours are fractionated. C. O. HARVEY.

Distillation of oils. R. E. HUMPHREYS, ASSR. to STANDARD OIL Co. (U.S.P. 1,647,629, 1.11.27. Appl., 31.3.22).—By distilling under pressure a hydrocarbon oil having an initial b.p. above the b.p. range of gasoline, a distillate containing gasoline and heavier fractions is obtained and is fractionated, the heavier fractions being again distilled under a pressure of over 300 lb./sq. in. C. O. HARVEY.

Continuous cracking and fractionation of hydrocarbons. A. C. SPENCER, ASSR. to STANDARD DEVELOPMENT Co. (U.S.P. 1,648,967, 15.11.27. Appl., 3.7.23).—A continuous stream of the oil to be treated is heated to cracking temperature and allowed to vaporise. The vapours are fractionally condensed as gasoline, kerosene, and a fraction heavier than kerosene. The last-named fraction is first removed and the others are separated by introducing the remaining vapours into a system of progressively-cooled zones at about its middle point. R. C. ODAMS.

Treatment of cracked hydrocarbon distillation products. H. BLUMENBERG, JUN. (U.S.P. 1,649,384, 15.11.27. Appl., 14.2.27).—The colour and odour of cracked gasoline are removed by treatment with aluminium chlorosulphate. H. ROYAL-DAWSON.

Treatment of hydrocarbon oils for use in internal-combustion engines. R. HENSE (F.P. 618,302, 9.3.26).—The oil (100 pts.) is scrubbed successively with nitric acid and sulphuric acid, and, after washing with water, is mixed with 8000 pts. of petroleum, 1500 pts. of heavy benzine, 400 pts. of light benzine, and 5 pts. of ammonium nitrate, and the mixture is distilled. L. A. COLES.

Recovery of readily volatile hydrocarbons from gases obtained in cracking and hydrogenating processes. P. WÜRTH (F.P. 616,582, 9.10.25).—Benzene is recovered by compressing the gases and then allowing them to expand. L. A. COLES.

Purification of light oils. A. RIEBECK'SCHE MONTANWERKE A.-G. (G.P. 442,597, 31.10.24).—Light oils recovered from gases obtained in the distillation of lignite and coal are treated successively with a halogen or a substance yielding a halogen, and an alkali, and are then fractionated by distillation. L. A. COLES.

Determination of the water content of insulating oils. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of W. ESTORFF and W. NAGEL (G.P. 442,946, 20.2.24).—The water is determined by measuring the volume of hydrogen generated on treating the oil with an alkali metal, a suitable quantity of a solvent being added to remove condensation products formed on the surface of the metal. L. A. COLES.

Treatment of clays to adapt them for decolorising and deodorising oils. J. S. POTTER, ASSR. to S. W. SHATTUCK CHEMICAL Co. (U.S.P. 1,649,366, 15.11.27. Appl., 15.8.25).—The clay is mixed with sulphuric acid and heated to decompose the greater part of the sulphates, and, after further heating to get rid of acid fumes, the substances soluble in hydrochloric acid are removed. H. ROYAL-DAWSON.

Coal pulverising machine. C. E. BLYTH, ASSR. to A. HERBERT, LTD. (U.S.P. 1,649,148, 15.11.27. Appl., 13.4.26. U.K., 16.5.25).—See B.P. 255,187; B., 1926, 733.

Gas producer. W. CLIMIE (U.S.P. 1,650,187—8, 22.11.27. Appl., [A] 15.6.22, [B] 24.11.24. U.K., [A] 15.6.21).—See B.P. 184,323; B., 1922, 740 A.

Coke-oven doors. C. STILL (B.P. 267,881, 20.10.26. Ger., 22.3.26).

Refractories for retorts (B.P. 279,201).—See VIII.

Leather oil (B.P. 255,908).—See XV.

III.—ORGANIC INTERMEDIATES.

Calcium citrate. MELIS.—See VII. **Dehydration of alcoholic liquids.** BRUN.—See XVIII.

PATENTS.

Manufacture of carbon disulphide. J. and A. KOMLOS and E. F. ENGELKE, Assees. of A. VON VAJDAFFY (B.P. 265,994, 11.2.27. Ger., 16.2.26).—Hydrocarbons, particularly acetylene, are allowed to react with sulphur at 380–450°, or with inorganic sulphides, e.g., iron pyrites, alone or with free sulphur at temperatures above 500° to produce carbon disulphide. Spent gas-purifying materials may be used as a source of sulphur, and the hydrogen sulphide evolved in the reaction

is absorbed by gas-purifying material to be returned to the process as a source of sulphur. W. G. CAREY.

Manufacture of organic acids. I. G. FARBENIND. A.-G., Assees. of G. and R. WIETZEL (G.P. 442,125, 10.6.23. Cf. B.P. 254,819; B., 1926, 721).—The following is additional. The reactions proceed also at ordinary pressure, but more slowly; *e.g.*, methyl alcohol vapour and carbon monoxide exposed to ultra-violet rays or led over barium and potassium carbonates at 500° give methyl formate and high-molecular products. Methyl formate and other low-boiling esters are obtained with a titanous acid catalyst at 140° under 500 atm. pressure. C. HOLLINS.

Reduction of aromatic nitro-compounds [production of iron oxide pigment]. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 279,283, 21.4.26).—When more than the usual 6 pts. of hydrogen chloride are used in the reduction of 100 pts. of nitrobenzene (*e.g.*, 18 pts.) the iron oxide by-product is obtained as a deep black, very finely-divided powder, having increased value as a pigment. C. HOLLINS.

Manufacture of intermediate products for dyes. SOC. CHEM. IND. IN BASLE (GES. F. CHEM. IND. IN BASEL) (B.P. 260,623, 30.10.26. Switz., 31.10.25).—A halide of a naphthylthioglycollic acid is halogenated; *e.g.*, *β*-naphthylthioglycollyl chloride, prepared from the acid and phosphorus pentachloride in chlorobenzene, is treated, without isolation, with sulphuryl chloride in presence of ferric chloride at 0–5°, to give 1-chloro-*β*-naphthylthioglycollyl chloride. C. HOLLINS.

Process of chlorination employing contact material having large surface. RHEINANIA-KUNHEIM VER. CHEM. FABR. A.-G., Assees. of H. FRITZWEILER, B. C. STUER, and W. GROB (G.P. 443,020, 5.4.24).—Natural hydrated metallic oxides which have been dehydrated by heating at a dull red heat and, if desired, under reduced pressure, are employed as contact material. J. S. G. THOMAS.

Stable mixture yielding salts of halogenosulphonamides. H. GÜNZLER and G. WESENBERG, Assrs. to WINTHROP CHEMICAL CO., INC. (U.S.P. 1,650,124, 22.11.27. Appl., 8.9.25. Ger., 18.10.24).—See B.P. 241,579; B., 1926, 565.

Dehydration of alcohol (B.P. 268,728 and F.P. 615,732). **Butyl alcohol and acetone** (B.P. 278,307).—See XVIII.

IV.—DYESTUFFS.

PATENTS.

Manufacture of new azo dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 279,146, 20.5.26).—2:3-Hydroxynaphthoic arylamides are coupled in substance, on the fibre, or on a substrate, with diazotised amines of the type, $\begin{matrix} (1) \text{NH}_2 \\ (2) \text{R} \end{matrix} > \text{Ar}-\text{R}^1$ (5), where Ar is any tervalent benzene residue, and of R and R¹ one represents an alkylsulphonyl, aralkylsulphonyl, arylsulphonyl, acyl, carbonamide, or sulphonamide group, whilst the other represents a halogen, alkyl, aryl, ether, or thioether group. Amines of the type, $\begin{matrix} (1) \text{NH}_2 \\ (3) \text{NHR} \cdot \text{CO} \end{matrix} > \text{C}_6\text{H}_3 \cdot \text{OR}^1$, are excluded. Special fastness to bowling is claimed. Examples are 3-amino-4-

methoxydiphenyl sulphone → 2:3-hydroxynaphthoic *o*-phenetidine (red); 3-amino-4-methyldiphenyl sulphone → 2:3-hydroxynaphthoic α - or β -naphthylamide (bluish red); 3-amino-4-phenoxybenzophenone → 2:3-hydroxynaphthoic α -naphthylamide (wine-red); 3:3'-diamino-4:4'-dimethoxydiphenyl sulphone → 2:3-hydroxynaphthoic *m*-chloroanilide (claret); *o*-toluidine-4-sulphonmethylamide → 2:3-hydroxynaphthoic α -naphthylamide (bluish-red) or *o*-phenetidine (red); 3-amino-4-methylbenz-*o*-chloroanilide → 2:3-hydroxynaphthoic anilide (bluish-red); 4-chloro-3-aminobenzanilide → 2:3-hydroxynaphthoic β -naphthylamide or 4-chloro-*o*-anisidine (yellowish-red). A table of 96 other couplings is given. The preparation of 3-amino-4-methoxy- and 3-amino-4-phenoxy-benzophenones from 4-chloro-3-nitrobenzophenone; of 2-amino-4-tolyl ethyl and benzyl sulphones from *o*-nitrotoluene-*p*-sulphinic acid; of 4-chloro-3-aminodiphenyl sulphone from 4-chloro-3-nitrobenzenesulphonyl chloride; of 3-amino-4-ethoxydiphenyl sulphone from 4-chloro-3-nitrodiphenyl sulphone; and of *o*-phenetidine-4-sulphondimethylamide from 4-chloro-3-nitrobenzenesulphonyl chloride, is described. C. HOLLINS.

Manufacture of a yellow azo dye [for acetate silk]. I. G. FARBENIND. A.-G. (B.P. 270,352, 3.5.27. Ger., 3.5.26).—*p*-Nitrodiazobenzene is coupled with 4-nitro-*m*-phenylenediamine. The product gives non-phototropic fast golden-yellow shades on acetate silk. C. HOLLINS.

Manufacture of monodiaz compounds of 1:4-diaminoanthraquinonemono- or di-sulphonic acids. I. G. FARBENIND. A.-G. (B.P. 264,879, 24.1.27. Ger., 25.1.26).—Diazotisation of 1:4-diaminoanthraquinone-2-sulphonic acid or its derivatives (*e.g.*, the 2:6 or 2:7-disulphonic acid) even in presence of excess of nitrous acid gives only a diazo (not a tetrazo) compound. The diazo compound may be converted into a chloro-compound by Sandmeyer's method, and thus provides an alternative route to dyes of the Alizarin Direct Blue A type. C. HOLLINS.

Manufacture of thiazole derivatives of 1:4-naphthaquinone. I. G. FARBENIND. A.-G., Assees. of A.-G. F. ANILIN-FABR. (B.P. 262,141, 29.11.26. Ger., 28.11.25).—2-Amino-3-thiol-1:4-naphthaquinone, prepared by the action of sodium sulphide on the 3-chloro-compound, is condensed with an aldehyde to give an intermediate product which is very rapidly converted by atmospheric oxygen into a naphthathiazole. The intermediate and the final products may be vatted with alkaline hyposulphite and give the same shades on cotton. Benzaldehyde yields an intermediate compound, decomp. 225–230°, and a final thiazole, m.p. 243°, which dye cotton a greenish-yellow, fast to chlorine and soap. α -Naphthaldehyde (reddish-yellow), *ar*-tetrahydro- α -naphthaldehyde (greenish-yellow), *p*-aminobenzaldehyde (violet), *p*-dimethylaminobenzaldehyde (reddish-blue), *o*-chloro-*p*-dimethylaminobenzaldehyde (violet-red), and half-molecular proportions of glyoxal (pale yellow) or terephthalaldehyde (yellow) give similar vat dyes. Sulphonated aldehydes yield acid wool colours. For the preparation of *ar*-tetrahydro- α -naphthaldehyde, 1-amino-methyl-5:6:7:8-tetrahydronaphthalene (B.P. 249,883;

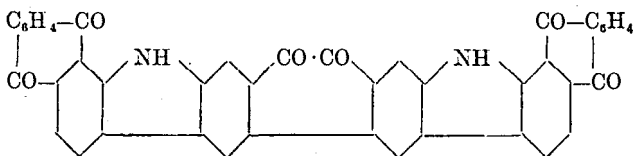
B., 1927, 572) is condensed with 1-chloro-4-nitrobenzene-2-sulphonic acid and the product is oxidised with chromic acid.

C. HOLLINS.

Production of stable preparations of vat dyes. I. G. FARBENIND. A.-G., K. F. MAENNCHEN, and F. HÜMMERICH (B.P. 278,890, 21.10.26. Addn. to B.P. 259,999; B., 1927, 869).—A mixture of dry, powdered caustic alkali and starch is used in place of the alkali-starch of the prior patent. Any absorption of moisture from the air at once leads to the formation of the innocuous alkali-starch compound.

C. HOLLINS.

Manufacture of new vat dyes of the anthraquinone series. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 279,003, 12.5.26).—A monobenzoyldiaminoanthraquinone containing a free α -amino-group is condensed with a halogenated phenanthraquinone in presence of an acid condensing agent (aluminium chloride, chlorosulphonic acid, sulphuric acid) at low temperature (15–45°). The secondary amine first formed undergoes further condensation to a carbazole derivative, this conversion being greatly facilitated by the presence of the benzamido-group. 2:7-Dibromophenanthraquinone gives with 1-amino-5-benzamidoanthraquinone a yellowish-brown, with 1-amino-4-benzamidoanthraquinone a reddish-brown, vat dye, the products being probably dibenzamido-derivatives of the carbazole:



C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 278,771, 12.5.26).—1-Aminoanthraquinone is condensed with a halogenated phenanthraquinone (2:7-dibromophenanthraquinone) as in B.P. 9888 of 1909 (B., 1910, 414), and the copper-red vat dye so obtained is treated at 150–180° with aluminium chloride in pyridine to form a reddish-brown vat dye having probably a carbazole structure (formula in preceding abstract).

C. HOLLINS.

Manufacture and use of vat dyes of the benzanthrone series. BRIT. DYESTUFFS CORP., LTD., A. SHEPHERDSON, and S. THORNLEY (B.P. 279,205, 26.8.26).—Benzanthrone and its non-sulphonated derivatives (except dibenzanthrone) are condensed with hydroxylamine in presence of sulphuric acid and ferrous sulphate. Fusion with alkali converts the products into grey to black vat dyes. Examples are benzanthrone and methylbenzanthrone.

C. HOLLINS.

Dyes and dyeing [vat dye of the dibenzanthrone series]. R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 278,834, 4.8.26).—The very pure dibenzanthrone obtained from dibenzanthronyl by the methods of B.P. 251,313 or 278,112 (B., 1926, 576; 1927, 903) is halogenated to give reddish-blue vat dyes having good fastness properties. Dichloro-, trichloro-, tetrachloro-, and dibromo-dibenzanthrones are described.

C. HOLLINS.

Manufacture of dyes and intermediates [dibenzanthronyls]. R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 278,496, 22.4.26; cf. B.P. 251,313; B., 1926, 576).—The method of the prior patent for the oxidation of benzanthrone to dibenzanthronyl and hydroxybenzanthrone is extended to substituted benzanthrone. 9-Chlorobenzanthrone, m.p. 183–185° (Scholl, Seer, and Daimler, A., 1922, i, 258), gives 9:9'-dichloro-4:4'-dibenzanthronyl, m.p. above 300°. The condensation of 2-chloroanthraquinone with glycerol yields a mixture of 4-chlorobenzanthrone, m.p. 189–190.5° (which cannot be oxidised to a dibenzanthronyl), and two isomerides, m.p. 130–134° and 148–150°, respectively, in which the 4-position is free, and which therefore give on oxidation *dichlorodibenzanthronyls*; they are presumably the 5- and 10-chlorobenzanthrones. Two isomeric chlorobenzanthrones, m.p. 180.5–181.5° and 154–160°, respectively, are obtained from 1-chloroanthraquinone; these yield *dichlorodibenzanthronyls* on oxidation. 9-Methylbenzanthrone (Scholl and Seer, A., 1913, i, 57) is converted into 9:9'-dimethyl-4:4'-dibenzanthronyl, but purified 4-methylbenzanthrone, m.p. 202° (prepared from 2-methylanthraquinone), does not react. By crystallisation from nitrobenzene and extraction of the more soluble products with benzene, isomeric methylbenzanthrone, which are oxidisable to *dimethyldibenzanthronyls*, are obtained from the condensation product of 2-methylanthraquinone with glycerol. The hydroxylated benzanthrone obtained in all these examples may be alkylated and converted into vat dyes by alkaline fusion.

C. HOLLINS.

Manufacture of azo dyes. W. NEELMEIER and T. NÖCKEN, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,650,090, 22.11.27. Appl., 7.5.26. Ger., 12.5.25).—See B.P. 252,182; B., 1927, 808.

Colour bases for varnishes (B.P. 278,765).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Determination of the amount of weighting in piece-weighted silk crêpe-de-chine fabrics. W. WELTZIEN and P. FOWINKEL (Textilber., 1927, 8, 157–158).—The methods usually employed are compared and criticised, the most satisfactory being that in which the weighted silk is extracted with cold 7% or warm 1.5% hydrofluoric acid and the loss of weight taken as a measure of the weighting substances present. When the degree of weighting is determined by the "nitrogen" method, the pure silk in the weighted crêpe fabric should be considered to contain 17.7% N, and to have suffered a loss of 30% in weight (not the usual 24%) during the previous operations of degumming and creping.

A. J. HALL.

Determination of the amount of weighting of silk crêpe-de-chine fabrics. W. ALTERHOFF (Textilber., 1927, 8, 367–368).—In a method for determining the amount of inorganic weighting in coloured (black excepted) silk materials, which is accurate and rapid, 1 g. of the silk crêpe-de-chine fabric is treated for ½ hr. at 60–70° with a solution containing 5–6 g. of oxalic acid per 100 c.c., then washed three times with hot water, similarly treated with a 2% solution of oxalic

acid, washed once, passed twice (20 min. each time) through a 2.5% solution of sodium carbonate at 60–70°, rinsed with cold water, air-dried, and weighed; the amount of weighting is measured by the loss of weight.

A. J. HALL.

Self-recording strength tester [for fibres]. F. RÜHLEMANN (Papier-Fabr., 1927, 25, 577–581).—The apparatus described is used for determining the breaking load, extension, and elasticity of fibres much more fragile than the ordinary textile fibres, and of stretching lengths between 0.5 and 15 mm. The fibre is mounted in a frame made from cinematograph film and suspended between one end of a balance beam and a fixed clamp in the base board. A loading pan is attached to a spring balance on the other end of the beam, and, after melting through the sides of the frame, the fibre is submitted to continuous loading, without shock, by the automatic raising of a burette, to the lower end of which a flexible tube is attached and through which water is delivered at a constant rate to the loading pan, thus maintaining a constant and regulated loading velocity. The quantity of water necessary to break the fibre gives the breaking load. The whole process of loading and extension is observed on an opaque camera screen through a specially constructed microscope, and on replacing the screen by a suitable box provided with a slit, past which film or photographic paper is slowly drawn, photographic records are obtained. The diagrams thus automatically produced show not only the loading process and the break, but also the extension and elasticity of the material examined. Further advantages of the apparatus are that stopping of the loading is effected instantaneously, and that elasticity may be measured during either loading or unloading of the fibre.

B. P. RIDGE.

Experimental yarn-sizing plant and some results obtained therewith. G. F. NEW (J. Text. Inst., 1927, 18, 303–310 T).—The size is mixed in a water-jacketed copper vessel, water from the jacket being pumped continuously through both the hollow sizing rollers and the jacket of the size box in order to maintain a constant temperature throughout the operation. The small-scale machine is so designed that all variables are adjustable, and can be maintained at any constant value. Above a certain temperature limit, which varies for different starches, and is usually well below 100°, no great difference is found in the character of the size produced by cooking at various temperatures, whilst the duration of cooking within the range $\frac{1}{4}$ –6 hrs. has but little effect. Concentration of the starch has an effect such that the protective power of the resulting size, as measured by wear tests on the yarn, is generally proportional to this concentration. Admixture with the size of oils and fats produces a decrease, and of glycerin an increase, in protective power, but if the oils and fats are applied to the surface of dry, sized yarn, a marked improvement is observed. Of the materials examined, paraffin wax and stearine have the greatest effect.

B. P. RIDGE.

Viscose. IV. The viscose film (cellophane). M. NUMA (J. Cellulose Inst., Tokyo, 1927, 3, 235–249).—In the preparation of films from ripened viscose the

permanence of the qualities of the film is perfectly satisfactory if a cellulose of high quality is used, but with low-quality wood pulp a marked degradation of the film may take place after a year. Under the conditions adopted the toughest films were obtained when the sulphurised viscose product was dissolved in caustic soda of 7% concentration. The quantity of ash, ranging between 0.2 and 1.0%, had no influence on the mechanical properties of the film. Within a certain limit of moisture content the tensile strength and elongation increase regularly with the thickness of the film, but with higher moisture the increase in elongation is no longer proportional to the increase in thickness. Increase in moisture decreases the tensile strength and increases elongation, but there is no definite relation. Above 12% of moisture these effects are strongly marked. Generally, if the film is comparatively thin, the influence due to a variation of 1–2% in moisture is negligible. The moisture content of a film of medium softness is 9–12% and independent of the thickness; a soft film has higher tensile strength and lower elongation than a harsh one, apart from the moisture content. The mechanical properties of the film depend chiefly on the physical properties rather than on the chemical properties of the pulp, although the colloidal condition plays a part. The higher the intensity of colour of the pulp in cuprammonium solution, the better are the mechanical properties. It is advantageous to dry the film before desulphurising and bleaching; the presence of free fibres in the film is stated to be advantageous from the point of view of strength and elongation, though affecting its transparency and lustre.

J. F. BRIGGS.

Hydration of cellulose in papermaking. J. STRACHAN (Proc. Tech. Sect. Papermakers' Assoc., 1926, 6, 139–167).—From a study of the behaviour of highly-compressed blocks of vulcanised rag paper, as representing in many ways the behaviour of individual fibres, evidence is adduced in support of the theory that the hydration of cellulose during beating is a purely physical phenomenon, cellulose exhibiting all the properties of an elastic gel whilst differing from other elastic gels in that it is porous and contains considerable quantities of adsorbed gases. Results are given showing the effect of pressure, vacuum, specific external surface, temperature, and dissolved substances on the velocity of imbibition of water by cellulose. Pressure has a greater accelerating action than vacuum, but the rate of increase of imbibition falls very considerably for pressures above 75 lb./sq. in.; increase in temperature causes dehydration, but in a negligible degree below 40°. Increase in specific external surface increases the velocity of imbibition proportionately, even when the increased surface is due in part to swelling of the fibres, as is the case after prolonged immersion; it is this increase in specific external surface, chiefly by fibrillation, that is responsible in a large degree for the hydration effect of beating. Alum, though showing initially an accelerating action, later retards imbibition; caustic soda accelerates or retards imbibition depending on the concentration; sulphuric acid is a more satisfactory accelerator, particularly at a concentration of 0.00125 molar. The Schopper-Riegler tester is unsatisfactory as an indicator of the degree of hydration of pulp, a more reliable method

being to determine the quantity of water retained by the pulp under a definite standard pressure.

D. J. NORMAN.

Mechanical [paper] pulp and its physical property of wetness. J. H. MOWAT (Proc. Tech. Sect. Papermakers' Assoc., 1926, 6, 197—204).—The importance of grinding temperature on the character and wetness of mechanical pulp and the equal importance, from the papermaker's point of view, of a knowledge of the degree of wetness are emphasised. Experiments have established that mechanical pulp after immersion in water for some days shows increased wetness. The addition of 1% of yeast (on the weight of fibre) to a 5% suspension of mechanical pulp caused after 18 hrs. at 18—21° an increase in wetness of 136%, dropping after 53 hrs. to a net increase of 91%. D. J. NORMAN.

Fuel for motor transport. IV. Power alcohol from grasses, etc.—See II.

PATENTS.

Washing or cleaning of wool. E. C. DUHAMEL, and COMP. GÉN. DES IND. TEXTILES (B.P. 273,755, 3.5.26. Fr., 4.5.25. Addn. to B.P. 256,635; B., 1926, 975).—The process of the original patent is modified in that the wool travels continuously or intermittently through one or more bowls with periodical stops for cleaning purposes. Provision is made for a continuous supply of fresh liquor, such as water or weak suint liquor, and for maintaining one or more baths of suint liquor of substantially high and constant concentration sufficiently clean for washing purposes by a purification treatment (other than settling), or by suitably pre-treating the wool. The process is applicable to wool-bearing skins and, in certain circumstances, to live sheep.

D. J. NORMAN.

Retting of flax and other fibres. BRIT. DYESTUFFS CORP., LTD., C. HOLLINS, and E. CHAPMAN (B.P. 279,583, 24.8.26).—Penetration of the fibre is assisted and fermentation begins more rapidly if, in the retting of flax, wetting-out or dispersing agents, namely, sulphonic acids of formaldehyde-phenol condensation products, alkylnaphthalenesulphonic acids, sulphonated isopropylated mineral oil fractions, ligninsulphonic acid, naphthenic acids, taurocholic acid, sulphonated higher aliphatic acids, etc. are added to the retting bath.

C. HOLLINS.

Material for treating vegetable fibres for use in the manufacture of paper. L., R., and G. SCHNEEBERGER (F.P. 617,826, 30.10.25).—The bleaching properties of the fibres are improved by softening them with a soap prepared from casein and resin, mixed with kieselguhr.

L. A. COLES.

Treatment of sulphate- and soda-pulp. R. COLLINS (U.S.P. 1,648,111, 8.11.27. Appl., 10.1.27).—The major portion of the spent cooking liquor is separated from the pulp in an undiluted form by drainage, and treated for recovery. The remainder of the liquor is removed by washing and the diluted liquor thus obtained is concentrated.

D. J. NORMAN.

Recovery of high-percentage acetic acid from solutions of acetylcellulose in acetic acid. VER. F. CHEM. IND. A.-G. (B.P. 268,778, 28.3.27. Ger., 1.4.26).—Concentrated acetic acid (90—95%) is recovered from

solutions of cellulose acetate in acetic acid, either before or after partial hydrolysis of the cellulose acetate to the stage of solubility in acetone, by subjecting the solution, initially at 90—100°, to distillation (without application of external heat) until the temperature falls to 50°. The solution may again be heated to 90—100° and the process repeated. Each of these treatments removes about 20% of the acetic acid without impairing the homogeneity of the solution, its spinning properties, or the character of the coagulated products.

D. J. NORMAN.

Manufacture of artificial cork. H. WADE. From ARMSTRONG CORK CO. (B.P. 279,190, 6.8.26).—Artificial cork blocks suitable for insulating purposes, particularly cold storage, are made by compressing cork particles in a mould and forcing through the compressed mass hot gases, e.g., hot air or steam or a mixture thereof, at 220—310°, to promote an exothermic reaction of an oxidising character whereby the cork particles become agglomerated into a compact mass. Suitable apparatus is described. [Statutory ref. to B.P. 26,074 of 1907.]

D. J. NORMAN.

Manufacture of paper. A. E. BARNARD and R. G. CASWELL, ASSRS. to W. B. PRATT, INC. (U.S.P. 1,648,838, 8.11.27. Appl., 30.7.23).—Paper of increased bursting strength is obtained by beating cellulose pulp in the presence of ammonia.

D. J. NORMAN.

Treatment of paper. E. B. EDDY CO., LTD., ASSEES. of H. SCHERBAK and A. LUTZ (Can. P. 264,999, 6.12.24).—Paper impregnated with glue solution is dried and treated with formaldehyde.

L. A. COLES.

Manufacture of composition paper or pulp board. J. H. MITCHELL (U.S.P. 1,648,237, 8.11.27. Appl., 4.3.27).—The board consists of sugar-cane bagasse fibres bound together by fibres obtained from the bagasse of sisal leaves.

D. J. NORMAN.

Loosening of vegetable fibres. J. BILLWILLER (U.S.P. 1,649,281, 15.11.27. Appl., 11.9.22. Switz., 13.7.22).—See B.P. 206,570; B., 1924, 90.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Bleaching of "back-greys" [in printing] stained with indanthrene dyes. M. M. TSCHILIKIN (Textilber., 1927, 8, 280—281).—Back-greys, after use for printing indanthrene dyes, become stained in colours very resistant to bleaching when afterwards subjected to a kier boil. Thus, Helindone Orange R yields a pinkish-orange stain; Thioindigo Red B a violet. The staining is due to dissolution of the loose dye on the back-grey in the alkaline kier liquor, which has a reducing action, so that dyeing of the back-grey in a pale shade then occurs. Staining is prevented, so that the back-greys may be satisfactorily bleached, by adding Leucotrope W to the kier liquor and first maintaining this for 15 min. at 50—100° and ordinary pressure. Alternatively stained back-greys may be decolorised by boiling for 15 min. at 100° in a liquor containing 5 g. of caustic soda and 0.03 g. of Leucotrope W per litre.

A. J. HALL.

PATENTS.

Production of Aniline Black on textile fibres. CALICO PRINTERS' ASSOC., LTD., L. A. LANTZ, and

R. WATSON (B.P. 279,164, 20.7.26).—Cotton fabrics are impregnated with the usual substances (*e.g.*, aniline, hydrochloric acid, sodium chloride, and an alkali ferrocyanide) for "prussiate" Aniline Black, dried, and subjected first to a dry heat of 110–180° for 5–45 sec., then heated in the presence of steam for 15–60 sec. at 100–180°, washed, and dried. The dry heat treatment promotes the formation of an Aniline Black which is ungreenable without the usual after-chroming treatment.

A. J. HALL.

Dyeing of textile materials. C. B. WHITE, Assr. to VIVATEX PROCESSES, INC. (U.S.P. 1,648,433, 8.11.27. Appl., 16.4.24).—Textile materials are coloured by impregnation with a metallic sulphide, and further rendered resistant to acid substances which may be formed subsequently by the action of air or other influences on the sulphide, by depositing within the materials an insoluble basic compound incapable of forming a sulphide in the presence of water.

A. J. HALL.

Dyeing and printing of acetylcellulose. H. Hoz, W. BERNOULLI, and A. LINK, Assrs. to J. R. GEIGY, Soc. ANON. (U.S.P. 1,650,275, 22.11.27. Appl., 22.7.25. Ger., 31.3.23).—See B.P. 231,897; B., 1925, 588.

Dyeing of furs (U.S.P. 1,649,502).—See XV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Oxidation of sodium amalgam by means of oxygen. A. FELDMANN (Giorn. Chim. Ind. Appl., 1927, 9, 455–456).—When sodium amalgam is vigorously shaken with pure oxygen in absence of water in a glass vessel the mass becomes pasty and heated. If alcohol also is present, acetaldehyde is formed, the alkali partially resinifying the aldehyde. Attempts to prepare peroxidised compounds from boric acid and baryta in this way proved unsuccessful. In an autoclave under a pressure of 10 atm., the reaction between sodium amalgam and oxygen yields sodium peroxide quantitatively. The industrial conversion of this into hydrogen peroxide has not yet been found possible.

T. H. POPE.

Oxides of iron, especially ferrous oxide. H. GROEBLER and P. OBERHOFFER (Stahl u. Eisen, 1927, 47, 1984–1988).—The purest preparation of ferrous oxide obtained by heating ferric oxide in a stream of carbon monoxide and carbon dioxide at 800° contained 99% FeO when only small quantities (1 g.) of material were used. By heating at 900° the preparation contained about 15% of metallic iron, about two thirds of which could be removed by treating the mixture with iodine solution; the residual ferrous oxide contained only 95% FeO. The m.p. of ferrous oxide, as determined by extrapolation from the results obtained with various impure specimens, is 1377°. Röntgenographic examination of oxides of iron prepared by reduction of ferric oxide at 800° shows that the limit of solubility of ferrous oxide in ferrosiferrous oxide is 5%; with more than 61% FeO the lattice structure of ferrosiferrous oxide is entirely replaced by that of ferrous oxide, which therefore appears to hold 39% Fe₃O₄ in solid solution. Ferrous oxide retains only traces of iron in solid solution.

A. R. POWELL.

Chemical utilisation of gypsum. G. GALLO (Giorn. Chim. Ind. Appl., 1927, 9, 405–410).—The only two successful processes for the chemical utilisation of gypsum consist (1) in the manufacture of sulphuric acid by the contact process from the sulphur dioxide obtained when a mixture of gypsum, silica, alumina, and coal is heated in a revolving cement furnace, the residual clinker being used for making cement, and (2) in passing ammonia and carbon dioxide into an aqueous suspension of powdered gypsum, the resultant products being calcium carbonate and ammonium sulphate. The author finds that, in presence of water, hemihydrated calcium sulphate and magnesium carbonate in about equimolecular proportions react at the ordinary temperature, a solution containing about 50 g. of magnesium sulphate per litre being obtained after 45 days. With dihydrated calcium sulphate the reaction is slower, and the resulting magnesium sulphate solution contains only 30–35 g. of the salt per litre. Other uses for magnesium sulphate are being sought. T. H. POPE.

Use of gypsum in the manufacture of ammonium sulphate. P. BAUD (Compt. rend., 1927, 185, 1138–1141).—The dried and sieved native gypsum (400 g.) containing 73.37% CaSO₄, 4.32% CaCO₃, and traces of alumina, iron, magnesia, and silica, is mixed with 70 g. of clay (SiO₂ 72.20%, Al₂O₃ 13.05%), 350 c.c. of commercial ammonia (32% NH₃), and 600 c.c. of water in an autoclave, the mixture being well stirred and a current of carbon dioxide bubbled through. The maximum yield of ammonium sulphate (96.6%) is obtained after 40 min. treatment at the initial and final temperatures of 70° and 48°, respectively, under a pressure of 750 g. The ammonia is recoverable from the mother-liquor, and the filtered paste when heated at 1550–1600° gives a useful clinker (SiO₂ 23.59%, CaO 66.77%, Al₂O₃ 4.28%, with traces of titanium, magnesium, and iron). This procedure ensures the complete reaction of the gypsum. J. GRANT.

Economic production of calcium citrate in a highly pure state. B. MELIS (Giorn. Chim. Ind. Appl., 1927, 9, 457–464).—The following process yields a perfectly crystalline calcium citrate with no free lime and no so-called brown extractive matters and containing 3% of organic salts, including soluble non-titratable citrate, and 70–72% of citric acid (theory, 73.6%). The lime used is first purified to remove the numerous undecomposed granules of oxide which are the cause of the resinoid condensation of the sugars of the agro and of the separation of the brown extractives. The lime is slaked in three communicating vessels at different levels, the coarser particles being allowed to settle. The agro is neutralised by the finer lime suspension, which is added gradually, the neutralisation being completed at boiling temperature and with continual stirring, so that long contact of the lime with the sugars present and precipitation of calcium salts other than the citrate are avoided. Preliminary filtration of the agro is unnecessary, the fine colloidal detritus present promoting the formation of finely crystalline citrate which is readily attacked by sulphuric acid.

T. H. POPE.

Nitrogen fixation by the high-tension arc. P. G.

COLIN and H. V. TARTAR (J. Physical Chem., 1927, 31, 1539—1558; cf. B., 1926, 485).—The effect of reduced pressure on the yield of fixed nitrogen with a constant velocity of gas molecules through the high-tension arc, together with the equilibria obtained in an arc under varying conditions, are studied on a laboratory scale. Under reduced pressure there is a decrease both in the concentration of nitric oxide and in the yield of fixed nitrogen per kw.-hr. The maximum equilibrium concentration of nitric oxide obtained over a wide range of pressure was dependent on the current passing, and the effect of variation in the velocity of the gases on this concentration was small. High concentrations of nitric oxide can be produced in an uncooled high-tension arc, the maximal concentrations for air (8.8%), for invert air (12.2%), and for equal volumes of oxygen and nitrogen (13.1%) being obtained at 100 mm. pressure. Water-cooling of the emergent gases is unnecessary. Equilibrium runs with air at atmospheric pressure without circulation gave a maximum concentration of 5.1% of nitric oxide with a current of 10—12 milliamp. The law of mass action holds approximately for the reaction between oxygen and nitrogen in the high-tension arc at pressures greater than 0.5 atm. L. S. THEOBALD.

Determination of carbon monoxide with iodine pentoxide. J. TAUSZ and K. JUNGSMANN (Gas- u. Wasserfach, 1927, 70, 1049—1051).—The gas mixture is passed in a slow stream from a water-jacketed burette through a series of wash bottles containing, respectively, potassium hydroxide, fuming and concentrated sulphuric acids, solid potassium hydroxide, and phosphorus pentoxide, and over iodine pentoxide (3—4 g.) maintained at 195° by means of a Pregl heating jacket containing aniline and a few drops of spindle oil. The reaction products—iodine and carbon dioxide—pass through a tube containing beads moistened with 10% potassium iodide, and finally through a wash bottle with baryta. The potassium iodide tube is thoroughly washed out with chloroform and potassium iodide, and the free iodine is titrated with 0.01*N*-thiosulphate. The carbon dioxide may be determined gravimetrically by converting the precipitated barium carbonate into sulphate. It is essential to pre-heat the iodine pentoxide to 195°, as below this temperature iodic acid is not completely dehydrated. W. T. K. BRAUNHOLTZ.

Determination of the carbon monoxide content of burnt gases from gas-burning appliances. GASINSTITUT. (Gas- u. Wasserfach, 1927, 70, 1052).—The method of Tausz and Jungsmann (preceding) gives excellent results, but, owing to the small carbon monoxide content of the gases, it is necessary to use at least 500 c.c. for each determination.

W. T. K. BRAUNHOLTZ.

Determination of the constituents of a gaseous mixture containing hydrogen sulphide, carbon dioxide, hydrogen arsenide and phosphide, and acetylene. M. WILMET (Compt. rend., 1927, 185, 1136—1138).—The hydrogen sulphide is first absorbed in a neutral solution of zinc acetate and the carbon dioxide then removed in potassium hydroxide. Arsine is then absorbed in a neutral 80% solution of cadmium acetate,

which will absorb 40 times its volume of the gas, and the phosphine and acetylene are finally determined by the reduction of a 30% solution of selenious anhydride, and by the alkaline potassium iodomercurate reagent of Lebeau and Damiens (A., 1918, ii, 81), respectively. Absorption occurs rapidly, except for arsine and acetylene, when portions of the gas must be tested separately from time to time. J. GRANT.

Deterioration of steels. VANICK.—See X. **Chromium chloride solutions.** STIASNY and GRIMM.—See XV.

PATENTS.

Manufacture of hydrocyanic acid. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 279,530, 26.6.26).—Formamide vapour is completely converted into hydrocyanic acid and water without the production of carbon monoxide and ammonia by heating at 400—450° under diminished pressure, in the absence of other gases, with dehydrating catalysts, *e.g.*, natural or artificial zeolites, calcium chloride, or carbonates of the alkali metals.

W. G. CAREY.

Concentration of phosphoric acid by evaporation. E. L. LARISON, Assr. to ANACONDA COPPER MINING Co. (U.S.P. 1,648,137, 8.11.27. Appl., 30.1.26).—In the concentration of crude phosphoric acid solutions containing fluorides, corrosion of the apparatus is avoided by maintaining therein during the process a solution of the acid saturated with alkali fluosilicates; the deposited fluosilicate crust acts as a protective agent.

H. ROYAL-DAWSON.

Manufacture of potassium or potassium sodium cyanide from calcium cyanamide. DEUTSCHE GOLD- u. SILBER-SCHNEIDANSTALT VORM. ROESSLER (G.P. 443,455, 24.11.22. Addn. to G.P. 442,966; B., 1927, 937).—Calcium cyanamide is fused with potassium chloride or with a mixture of sodium and potassium chlorides, the mass extracted with water, and the lime removed by addition of an alkali salt of an acid which forms an insoluble calcium salt.

A. R. POWELL.

Maintaining the stability of dibasic calcium hypochlorite. I. G. FARBENIND. A.-G. (B.P. 276,307, 10.6.27. Ger., 18.8.26).—The stability of the compounds described in B.P. 188,662 (B., 1923, 656 A), is maintained by keeping the hypochlorites in a "centrifuge-moist" state, *i.e.*, with a degree of moisture between 5 and 15%.

H. ROYAL-DAWSON.

Production of aluminium chloride. H. BLUMENBERG, JUN. (U.S.P. 1,649,383, 15.11.27. Appl., 31.8.26).—A mixture of aluminium sulphate and potassium chloride is heated in a closed chamber in the presence of a reducing agent to such temperature that aluminium chloride is formed without volatilising the potassium chloride; the vapours of aluminium chloride so formed are condensed.

H. ROYAL-DAWSON.

Desiccation [dehydration] of molten metal chlorides. W. MOSCHEL and P. SIEDLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,650,531, 22.11.27. Appl., 11.2.26. Ger., 16.2.25).—The molten hydrated metal chloride is treated with calcium carbide.

H. ROYAL-DAWSON.

Production of barium silicofluoride. H. S. McQUAID, Assr. to GRASSELLI CHEMICAL Co. (U.S.P.

1,648,143, 8.11.27. Appl., 22.11.26).—A non-alkaline barium compound is acted upon by sodium silicofluoride.

H. ROYAL-DAWSON.

Working-up of fluorides containing silicic acid. RÜTGERSWERKE A.-G. (B.P. 271,816, 25.1.27. Ger., 31.5.26).—Fluorides containing silicic acid are decomposed with acid in wooden vessels yielding a mixture of hydrogen fluoride and silicon fluoride. If aluminium fluoride is required the mixture is treated with an aqueous suspension of aluminium hydroxide or kaolin, silicic acid being precipitated. Sodium fluoride and sodium fluosilicate are obtained by treating the mixture with an aqueous solution of soda or a sodium salt.

W. G. CAREY.

Mining of soluble boron compounds. H. BLUMENBERG, JUN. (U.S.P. 1,649,385, 15.11.27. Appl., 16.2.27).—Steam and air are conducted to the bottom of a shaft to the deposit, whereby the water of condensation leaches out the soluble contents and the liquid formed is raised to the ground surface.

H. ROYAL-DAWSON.

Production of carbonic acid gas. B. LUNDIN (B.P. 269,478, 24.11.26. Swed., 17.4.26).—Sodium bisulphate is dissolved in water contained in a gas generator, or a solution of the salt is introduced into the generator, and an equivalent amount of solid sodium bicarbonate is added at a suitable rate by a screw conveyor; the resulting sodium sulphate liquor is removed and crystallised as Glauber's salt.

W. G. CAREY.

Separation of phosphorus from gases containing phosphorus. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 279,710, 11.4.27).—Gases containing phosphorus are cooled below the dew point of phosphorus, and water or molten phosphorus as rapidly moving drops is projected into the fog of phosphorus by a centrifugal washer of the Theisen or Ströder type. Two or more washers may be arranged in series, the first being operated at a temperature above the m.p. of phosphorus, the others below that temperature.

W. G. CAREY.

Production of a gas of uniform sulphur dioxide content. H. HOWARD, ASST. to GRASSELLI CHEMICAL Co. (U.S.P. 1,650,358, 22.11.27. Appl., 28.8.24).—Gas containing sulphur dioxide is mixed with sulphur vapour in quantity inversely proportional to its sulphur dioxide content, and the mixture burnt.

M. E. NOTTAGE.

Delaying the ignition of hydrogen phosphide resulting from the action of water on calcium phosphide or other phosphide. F. HEBLER (B.P. 279,751, 1.7.27).—The calcium phosphide is coated with a fused mass, e.g., potassium dichromate, pyrophosphate, borax, etc., in a rotating drum, or with water-free solutions of cellulose ester or other film-forming substances. The retardation is adjusted by a coating of greater or less solubility or permeability.

W. G. CAREY.

Apparatus for manufacture of lead oxide. H. WARING, ASST. to ASSOCIATED LEAD MANUFACTURERS, LTD. (U.S.P. 1,649,428, 15.11.27. Appl., 26.1.26. U.K., 30.1.25).—See B.P. 254,352; B., 1926, 744.

Recovery of thiocyanates (U.S.P. 1,648,224).—See II. **Carbon disulphide** (B.P. 265,994).—See III. **Recovery of acetic acid** (B.P. 268,778).—See V.

VIII.—GLASS; CERAMICS.

Analysis of soda-lime glass. G. E. F. LUNDELL and H. B. KNOWLES (J. Amer. Ceram. Soc., 1927, 10, 829—849).—Results of the analysis of a standard soda-lime glass distributed by the Bureau of Standards to thirteen analysts are summarised in tabular form. A general tendency towards high results, particularly with the alkaline earths and alkalis, was indicated. The accurate or "umpire" method of analysis of such a glass was then considered and detailed procedure laid down. Determinations so treated were loss on ignition, silica, iron oxide, alumina, titania, zirconia, lime, magnesia, soda, potash, manganese oxide, sulphur trioxide, arsenious and arsenic oxides, and chlorine. Fairly accurate shortened routine methods were also given.

A. COUSEN.

Changes in optical glasses. P. NICOLARDOT (Rev. gén. Colloid., 1927, 44, 592—598).—A detailed description is given of the "dimming test," used by Elsdon, Roberts, and Jones (B., 1919, 721 A) for determining the degree of adsorption of different glasses. The results obtained by this method for a number of glasses are in fair agreement with those obtained by the iodococin test.

L. L. BIRCHUMSHAW.

Extending the life of chemical glassware. J. T. LITTLETON JUN., and G. A. DASNEY (Ind. Eng. Chem., 1927, 19, 1271).—The production of strain, by subjection to extreme degrees of heat, is evidenced in a photograph of a Kjeldahl flask taken in polarised light. Three broken circles of light near the bottom of the flask show where the heat, by slightly softening the glass, had produced a permanent strain. Similar strains, later causing breakage under conditions of only moderate service, are produced by evaporating the contents of a flask to dryness over a flame, or by using an intense pointed flame under a flask containing a liquid. The danger of scratches on the inner surfaces of glass vessels is emphasised. During heating, the hotter, outer surface tends to stretch the inner surface, and the stresses at the sharp angle at the bottom of the scratch are increased, thus increasing the liability to fracture.

E. H. SHARPLES.

Constitution of clay-mud. H. CHATLEY (Inst. Civil Eng., 1927, No. 52, 3—15).—The characteristic physical properties of clay as compared with wet sand of similar composition—considerable tensile strength and apparent cohesion, also its impenetrability to water—are the consequence of the fineness of the particles which renders molecular forces important. The properties are best shown when there is a sufficiency of colloid particles to cover and cement the larger ones. Terzaghi's theory of clay structure is discussed. The author has investigated quantitatively the settlement of clay particles from a fresh-water suspension by addition of sea salt, a phenomenon due to the neutralisation of the charges on the colloid particles by the adsorption of oppositely charged ions. It was found that sea salt was three times as effective as pure sodium chloride in producing coagulation, this being due to the effect of bivalent calcium and magnesium ions. Alum is more than ten times as effective as salt. There is an optimum concentration of electrolyte for a given colloid concentration, as reversal of the charge takes place with an excess.

C. IRWIN.

PATENTS.

Manufacture of laminated glass. O. Y. IMRAY. From L. BARTELSTONE (B.P. 279,671, 21.12.26).—To the surfaces of a flexible sheet, such as celluloid or cellulose acetate, is applied a coating of pure vegetable oil having no solvent action on the film (*e.g.*, castor oil). The sheet is then sandwiched between two sheets of glass, the contact surfaces of which have been previously coated with a film of celluloid, adhesion being secured by applying a pressure exceeding 1 atm. for a short time while the temperature is substantially raised (*e.g.*, 150 lb./sq. in. for 8 min. and 110°). A. COUSEN.

Bleaching or decolorising of clay or other minerals. T. W. PARKER, and DARTMOOR CHINA CLAY Co., LTD. (B.P. 279,694, 28.2.27).—Clays or other minerals containing iron are decolorised by treatment with acid, either with or without a reducing agent, and with aqueous solutions of soluble salts. Such salts must neither be reducing agents nor their bases form coloured compounds with acidic radicals which are non-chromophoric; their acidic radicals also must not form insoluble iron compounds. Ammonium salts or salts of multivalent metals such as calcium, barium, or magnesium may be used, but aluminium salts (*e.g.*, the sulphate or an alum) are to be preferred. The salt may be added to the bleaching liquid before treatment or to the washing water subsequently used or first mixed with the clay suspension. A. COUSEN.

[Protection of] refractory bodies such as carbonising retorts. SOUTH METROPOLITAN GAS Co., and R. H. B. LAMPREY (B.P. 279,201, 21.8.26).—Those portions of a carbonising retort etc. which are exposed to the highest temperature are protected by a covering of highly aluminous material of plastic clay, calcined alumina, and grog formed into a plastic mass or as tiles. The surface is first painted with a mixture of the clay and sodium silicate to ensure adhesion. W. G. CAREY.

Zirconia-faced refractory. P. G. WILLETS, ASSR. to HARTFORD-EMPIRE Co. (U.S.P. 1,650,577, 22.11.27. Appl., 20.1.26).—A refractory body has a facing of substantially pure zirconium oxide which is partially interpenetrated by it and therefore united integrally with it. M. E. NOTTAGE.

Manufacture of tiles. V. LEFEBURE (U.S.P. 1,650,080, 22.11.27. Appl., 4.3.27. U.K., 7.12.25).—See B.P. 268,857; B., 1927, 443.

Apparatus for annealing glassware. BRIT. HARTFORD-FAIRMONT SYNDICATE, LTD., ASSEES. of E. O. HILLER (B.P. 273,724, 29.6.27. U.S., 1.7.26).

IX.—BUILDING MATERIALS.

Air-seasoning and conditioning of timber. F. M. OLIPHANT (Dept. Sci. Ind. Res., Forest Prod. Res., 1927. Spec. Rep. No. 1. 26 pp.).—The drying and seasoning of wood are complicated by the heterogeneous cellular structure. Wet green wood may contain double the weight of water compared with that of the dry wood, as free water in the cell cavities and water absorbed by the cell walls, the latter moisture causing considerable lateral shrinkage on evaporation. This shrinkage is by no means uniform, due to the presence of bands known

as wood rays, which restrict contraction of the adjacent cell walls. Hence "checking" and warping (cupping and twisting) result from internal straining. End-checking of sawn logs can be diminished by coating the ends with paint or a resin preparation; sometimes paraffin wax is used on the ends of high-class furniture timber. Case-hardening is caused by quick surface-drying and may give rise to cupping, and a further defect known as collapse is probably due to the rapid removal of water in the cells without a corresponding penetration of air, the cell walls thereby being drawn together. When air-seasoning, advantage must be taken of drying air currents, while avoiding undue shrinkage and a heavy decay by fungi. Illustrations of various methods of piling are given. In addition to reducing weight, increasing stability, and reducing the liability to decay, the seasoning of wood increases all the strength values except toughness. The prevailing poor reputation of kiln-dried wood is due to excessive drying below the fibre saturation point, together with the use of too high temperatures at the commencement of the "run." C. A. KING.

PATENTS.

Manufacture of cement mortar, artificial stone, concrete, etc. C. PICKSTONE (B.P. 279,355, 16.6.27).—In the manufacture of cement mortar etc. from Portland cement and sand, crushed bricks, etc., 5–15% of fine slate powder replaces an equal amount of the inert material, the ingredients being mixed in the dry state and then with water to which has been added $\frac{1}{4}$ – $\frac{1}{2}$ % of sodium silicate. W. G. CAREY.

Building cement or plaster. C. E. KRAUS (B.P. 253,929, 17.6.26. U.S., 18.6.25).—Clay, preferably containing at least 12% Al, is causticised with caustic soda solution, roasted at a temperature below that which will drive off all water of crystallisation, mixed with an alkaline earth containing a large percentage of calcium compounds, *e.g.*, dolomitic limestone, digested as slurry with sulphuric acid, and treated with more alkaline earth. The whole mixture is roasted at 100–315° depending upon the setting ability desired in the final product. W. G. CAREY.

Preparation of road-building material. O. L. DILLON, JUN. (U.S.P. 1,648,166, 8.11.27. Appl., 27.5.27).—The mineral aggregates are moistened with an alkaline solution, dried, and heated before admixture with bitumen. H. ROYAL-DAWSON.

Manufacture of porous materials [from sugar-factory residues]. A. MENAGER (B.P. 280,116, 20.5.27).—Caked residue from sugar refineries is placed in a mixing receptacle and covered with the mortar etc. to be treated, with which is incorporated agar-agar or emulsions of resins, tar, bitumen, asphalt, etc. to give a protective film and, together with the albumin, casein, gums, etc. in the residue, to increase the number and maintain the permanency of the gas bubbles in the final product. Sufficient acid is then added to the residue to liberate its carbon dioxide, and the whole agitated to produce a homogeneous mass. F. R. ENNOS.

Rendering [cement] surfaces fluid-tight. J. A. H. ITIER, ASSR. to SOC. LE TEXTILON CENTRAL (U.S.P. 1,649,592, 15.11.27. Appl., 12.2.26. Fr., 18.2.25).—See B.P. 247,976; B., 1927, 190.

Abrasive cement. H. O. KEAY, Assr. to LAURENTIDE Co., LTD. (U.S.P. 1,650,133, 22.11.27. Appl., 11.8.25).—See B.P. 260,171; B., 1926, 1015.

Preservation of wood etc. K. H. WOLMAN, F. PETERS, and H. PFLUG (B.P. 263,757, 6.11.26. Ger., 2.1.26).—See U.S.P. 1,622,751; B., 1927, 678.

Method and means for preparing a wet mixture of fibre and cementitious material. J. S. HANCOCK and W. H. MACMENIGALL (B.P. 280,456, 30.5.27).

Superheating slag (B.P. 271,087).—See X.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Equilibria in the reduction, oxidation, and carburization of iron. IV. R. SCHENCK [with A. DAHM, F. FARR, G. FINCK, W. HEMPELMANN, N. JUSCHKEWITSCH, (Fr.) H. NIPPERT, F. TRAUMANN, J. BÖKMANN, T. DINGMANN, R. FRICKE, W. KESTING, W. PRATJE, and A. SAVELSBERG] (Z. anorg. Chem., 1927, 167, 254—314; cf. A., 1927, 1030).—The composition of the mixture of carbon dioxide and carbon monoxide in equilibrium with metallic iron, and mixtures of ferrous oxide and different forms of carbon with or without the metal, has been determined at different temperatures. The composition of the gas phase when equilibrium is reached in the action of carbon monoxide on the metal corresponds with the equilibrium set up over oxoferrite and wüstite as solid phases, but when graphite or one of the other forms of carbon is present the concentration of carbon monoxide in the gas phase is rather greater. Small amounts of mercury cause an increase in the concentration of carbon monoxide in the equilibrium gas phase obtained starting from pure iron, probably because the amalgamation of the iron causes a reduction of the solubility of oxide in the metal, giving an oxoferrite poorer than normal in oxygen. It is found that in the equilibrium set up in the absorption of carbon monoxide by metallic iron the pressure of the gas phase at a given temperature is not constant, although its composition is, which points to the existence of a third solid phase, containing carbon, probably as a solid solution of carbide. Experiments in which cementite was heated in carbon monoxide show that this phase must also contain oxygen, and the name "oxoaustenite" is therefore suggested. In order to ascertain the relations between the three phases, mixtures of cementite with ferric oxide or ferrosferic oxide were heated at 600°, 650°, and 700°, the gas phase being removed and analysed when equilibrium had been attained, and this process repeated until no more gas was given off. At each temperature the curve connecting the pressure of the gas phase with the concentration of oxygen in the solid phase consists of two flat portions parallel to the concentration axis, and separated by a descending portion, and is terminated by another descending section. Until the commencement of the final stage, however, the composition of the gas phase remains unchanged, corresponding with the wüstite-oxoferrite equilibrium, the variations in the total pressure of the gas phase being due to the oxoaustenite. If the total reaction pressure is plotted against the composition of the gaseous phase, there is in each curve a middle portion parallel to the axis of pressure,

and corresponding with the wüstite-oxoferrite-oxoaustenite equilibrium, whilst the two extreme portions, which are concave to the axis of pressure, correspond with the bivariant equilibria set up with the solid phases wüstite and oxoaustenite, and oxoferrite and oxoaustenite, respectively. By means of the equilibrium curve for the system in which the solid phases are cementite and oxoaustenite, the field of existence of oxoaustenite can therefore be determined. Below 600°, four other univariant systems are also possible, viz., wüstite-cementite-oxoaustenite-gas, ferrosferic oxide-wüstite-cementite-gas, ferrosferic oxide-wüstite-oxoaustenite-gas, and ferrosferic oxide-cementite-oxoaustenite-gas. At 560°, wüstite disappears, and below this temperature only the last of these is therefore possible. Above 650°, the only bivariant systems, other than wüstite-oxoferrite-gas, which could be examined were oxoferrite-oxoaustenite-gas and cementite-oxoaustenite-gas, and data for these up to 800—900° have been obtained. An examination of the equilibrium set up between pure iron, or iron containing carbon, and carbon monoxide or a mixture of monoxide and dioxide at 800—1110°, makes it appear probable that Matsubara's supposed bivariant carburization equilibria (A., 1921, ii, 644) are actually tervariant oxoaustenite equilibria. The only case in which free carbon may exist in equilibrium in the solid phase in any of the equilibria examined seems to be when a mixture of amorphous carbon or finely-divided graphite with wüstite or an oxoferrite-wüstite mixture is heated at a temperature above 680°.

R. CUTHILL.

Equilibria in the reduction, oxidation, and carburization of iron. V. R. SCHENCK (Z. anorg. Chem., 1927, 167, 315—328; cf. preceding abstract).—The data obtained for the composition of the gas phase in equilibria in the system iron-carbon-oxygen have been used to construct a space model, isothermal sections of which are reproduced.

R. CUTHILL.

Superficial hardening of steel. G. CHARPY (Compt. rend., 1927, 185, 990—991).—Carburization of a steel, in which a superficial layer of cementite is produced, is distinguished from cementation, in which the carbon dissolves slowly in the iron in proportions increasing gradually with time. The optimum temperature of formation of carbides is 650°, since this strikes a balance between their rates of formation and decomposition, and the method is thus best applied to semi-hard steels, which at this temperature also acquire a maximum resilience. For pure iron and ordinary steels, gases containing large proportions of carbon dioxide, hydrocarbons, or cyanogen are recommended as carburising agents. For special steels, in which mixed carbides may be produced, hydrocarbons, cyanide or ferrocyanide baths, or molten potassium cyanide may be used.

J. GRANT.

Annealing of hardened steel, particularly at low temperatures. A. MERZ and C. PFANNENSCHMIDT (Z. anorg. Chem., 1927, 167, 241—253).—The changes occurring when quenched steels are heated have been investigated by means of differential heating curves. With a sample of steel containing 1.62% C, and quenched from 1100°, irregularities in the heating curve corre-

sponding with the decomposition of martensite and of austenite were observed, the actual temperatures at which these effects commenced depending, however, to a considerable extent on the rate of heating. Previously cooling the sample in liquid air caused the martensite effect to increase and the austenite effect to decrease. A sample of the eutectoid composition showed both changes if quenched from 1100°, but only the austenite change if chilled from 700°. A steel with 0.45% C quenched from 1100° did not exhibit the martensite change unless previously cooled in liquid air. In explanation of these results, the existence of two forms of martensite is suggested, one form, decomposing above 100°, being produced by quenching from high temperatures or by cooling austenite in liquid air.

R. CUTHILL.

Deterioration of structural steels in the synthesis of ammonia. J. S. VANICK (Trans. Amer. Soc. Steel Treat., 1927, 12, 169—194).—Specimens of ten carbon, nickel, or chromium steels were exposed for 4 months to an $N_2:3H_2$ gas mixture containing 8.3% NH_3 at 500° and 1500 lb./in.² pressure. All were attacked and the tensile strength and ductility were lowered. The carbon steels were readily decarburised and fissured. Nickel steels were deeply penetrated, but showed high residual strength. Chromium steels deteriorated more rapidly with high than with low carbon. Tungsten and nickel improved the resistance of chromium steels. Tests were also made for 10 months on 14 chromium-vanadium steels containing 0.2—0.6% C, 0.04—14.4% Cr, 0.07—0.65% V. Low carbon content was necessary for high resistance. At least 2.25% Cr was necessary to confine the decarburisation and fissuring to a uniform penetration. A higher chromium content improved the resistance but not proportionately. Two stainless steels with 13.5 and 21.1% Cr, respectively, were also tested and showed the least depth of deterioration. High-nickel-chromium steels are said to be most resistant, though difficult to forge and machine, but definite data are not given. Heat-treatment effected little or no improvement on the partly successful steels. Vanadium added no appreciable improvement. Decarburisation, fissuring, and the presence of combined nitrogen in the zones of attack are thought to be chiefly due to the action of "activated" hydrogen. A sharp increase in combined nitrogen was found in chromium steels. An explanation is attempted by a cyclic reaction theory of nitride formation and decomposition.

T. H. BURNHAM.

Hair cracks in steel rails. J. H. WHITELEY (Trans. Amer. Soc. Steel Treat., 1927, 12, 208—220).—Rails which were subject to deep-seated hair cracks after the heads had been sorbitised by cooling in air jets were found during etching to evolve a rapid stream of gas bubbles containing at least 50% CO, showing that the metal contained minute cavities filled with gas under pressure. It is considered that severe stresses resulted therefrom which might be the cause of local incipient cracks, the propagation of which was aided by the presence of foreign matter at the grain boundaries. The carbon monoxide might have been formed by the reaction between iron oxide and carbon during rolling.

T. H. BURNHAM.

Roman damascene steel. B. NEUMANN (Arch. Eisenhüttenwesen, 1927, 1, 241—244; Stahl u. Eisen, 1927, 47, 1695—1696).—Micrographic examination of Roman double-edged swords showed them to have been made by hammer-welding layers of steel of varying carbon content in such a way as to twist the layers into a V or W shape; subsequently, the cutting edges alone were hardened by a case-hardening process of some kind. The general absence of martensite in the structure of ancient steel articles is discussed and a theory advanced to account for it.

A. R. POWELL.

Effects of chromium and nickel in cast iron. R. S. POISTER (Amer. Foundrymen's Assoc., 1927, No. 27—28. Advance copy. 21 pp.).—Addition of 0.5% Cr with 1—2% Si to cast iron increases its strength and hardness; with 12.5—18% Cr the iron can be heated for a long time without oxidation, but is hard and brittle unless the carbon content is low (2.5%). Chromium up to 0.4% increases the strength of cast iron by formation of carbide instead of coarse graphite. Addition of nickel and chromium together (2—3:1) is preferable.

CHEMICAL ABSTRACTS.

Nickel changes properties of grey cast iron. D. M. HOUSTON (Foundry, 1927, 55, 399—401).—Nickel functions similarly to silicon in relation to carbon; it does not materially precipitate graphite below the upper critical range, but restricts the breaking-up of combined carbon.

CHEMICAL ABSTRACTS.

Work performance and bending strength of high-speed steels. W. OERTEL (Stahl u. Eisen, 1927, 47, 2036—2038).—The addition of cobalt to high-speed tool steels containing 0.6—0.7% C, 4.2—4.5% Cr, 17.0—18.4% W, and 1.3—1.9% V increases the cutting power very considerably, the best results being obtained with a high vanadium content. Equally good results are obtained with tungsten steels containing 2.9% Co and 1.9% V as with similar steels with 5.5% Co and 1.7% V. The maximum hardness combined with good elastic properties is produced by hardening at 1100—1200°. The bending strength of the steels hardened at 1325° falls to a very low value after tempering at 200°, rises somewhat again at 300°, falls again at 400°, then rises rapidly with increase of tempering temperature to 600—625°, within which range the maximum elasticity is obtained.

A. R. POWELL.

Influence of cobalt, vanadium, and manganese on the properties of tool steels. R. SCHERER (Arch. Eisenhüttenw., 1927, 1, 325—329; Stahl u. Eisen, 1927, 47, 2035—2036).—By increasing the manganese content of eutectoidal tool steels the maximum hardness obtainable by heat-treatment, the temperature range within which this hardness is obtained, and the temperature at which the steel is overheated are all lowered. Addition of vanadium increases the hardness to a maximum with 0.5% V. More vanadium, however, decreases the overheating temperature and the insensitive range. In the vanadium-silicon steels the hardness decreases with an increase in the quantity of the alloying elements, whereas in cobalt steels the hardness is almost independent of the cobalt content. The relatively large change of volume which plain carbon eutectoidal steels undergo on hardening is increased

by the addition of manganese and decreased by addition of vanadium and of silicon. The magnitude of this change of volume is independent of the cobalt content, and is somewhat greater with cobalt steels than with silicon-vanadium steels. The cutting power of manganese steel increases rapidly with the manganese content, and that of cobalt steel with the cobalt content, but vanadium steel has a maximum cutting power with 0.4% V. Tests on various steels containing two or more of these alloying elements indicated that the best cutting power combined with the smallest change of volume on hardening and therefore the greatest ability to undergo repeated hardening treatments is obtained with the steel containing 0.9% C, 2.28% Co, and 0.43% V.

A. R. POWELL.

Cast-iron diagram of Maurer with varying rates of cooling. E. MAURER and P. HOLTZHAUSEN (Stahl u. Eisen, 1927, 47, 1805—1812, 1977—1984).—A series of cast irons containing 2.4—3.8% C, 0.8—1.0% Mn, 0.3% P, 0.1% S, and varying quantities of silicon were cast in chill moulds and in sand moulds heated at 250° and at 450° and, from an examination of the microstructure of the castings, the Maurer equilibrium diagrams for cast iron under the different casting conditions have been constructed (cf. B., 1924, 911). For chill-cast specimens the boundary of the pearlite field is displaced towards the right, the line meeting the axis of carbon content (1% C) at 2.7% Si instead of at 2.5% Si; at its upper end this line bends towards the right with more than 3.1% C. For specimens cast into hot moulds (450°), the boundaries of the pearlite field are displaced to the left, cutting the axis at 0.5% Si less than in the Maurer diagram, whereas for specimens cast into moulds at 250° the Maurer diagram holds good. The rates of cooling of the castings in sand moulds preheated at 250° and at 450° correspond with the rates of cooling of cylinders of 75 and 90 mm., respectively, under foundry conditions. From the results obtained with these, therefore, the diagram for castings having walls between 10 and 90 mm. thick has been constructed. The presence of graphite eutectic together with ordinary graphite in both sand and chill castings and the increasing coarseness of the constituents with slower rate of cooling show that the ordinary form of graphite is obtained by a secondary action. The bending strength and hardness of cast iron are reproduced in the form of tables, graphs, and space-model diagrams.

A. R. POWELL.

Cutting cast iron with a flame. I. C. FRITZ (Gas-u. Wasserfach, 1927, 70, 1048—1049).—Cast iron has hitherto presented difficulties to flame cutting, since it melts before it burns, and its oxide melts before the metal itself. This difficulty is overcome by a new type of oxy-acetylene burner, in which the oxygen is preheated by passing over nozzles placed behind the concentric heating and cutting nozzles, and which, by first burning the carbon in the cast iron, allows the remaining iron to be easily cut. For example, castings with walls up to 150 mm. thick can be cut in a short time, the cut having a width of 8—20 mm.

W. T. K. BRAUNHOLTZ.

Treatment of articles of ferrous alloys in solutions of complex phosphates as a protection against corrosion. J. COURNOT (Compt. rend., 1927,

185, 1041—1043).—Clean iron, or ferrous alloys, immersed in a bath of a boiling acid solution (3—4%) of iron and manganese phosphates, liberate a small quantity of hydrogen and then become coated with an adherent grey-black deposit of a secondary phosphate of iron, about 0.005 mm. thick. The bath, which should contain about 30 g. of salts per m.² of metal surface, produces no change in the physical or mechanical properties of the metal and no surface-hardening as in the case of cementation. The surface is a good base for any of the usual finishing agents (lacquers, enamels, etc.) and has a resistance to corrosion by air, fresh, sea, or carbonated waters, water vapour, vinegar, and 5% solutions of sodium carbonate, bicarbonate, or sulphite, equal to or higher than that produced by other protecting processes. On account of its close adherence to the metal it is also less vulnerable to localised corrosion.

J. GRANT.

Influence of an addition of chromium on the internal friction of reversible ferro-nickels. P. CHEVENARD (Compt. rend., 1927, 185, 1130—1132).—The addition of chromium to reversible ferro-nickels of the invar group extends the range of temperature in which the internal friction is relatively feeble. This effect, and its relation to the anomaly previously recorded (B., 1927, 279), are shown in a three-dimensional diagram.

J. GRANT.

Iron-chromium equilibrium diagram. P. OBERHOFFER and H. ESSER (Stahl u. Eisen, 1927, 47, 2021—2031).—The equilibria in the system iron-chromium have been determined by thermal analysis and Röntgenographic investigation of a series of alloys made from electrolytic iron and the purest chromium. The metals form a continuous series of solid solutions with a minimum m.p. at 1405° for the alloy with 14.5% Cr. The temperature of the A4 transformation decreases steadily with the addition of chromium at the rate of 26° for every 1% Cr up to 14% Cr. The A3 temperature falls to a minimum of 840° (heating) and 812° (cooling) with 8% Cr, then rises with further addition of chromium, the curve finally meeting the A4 curve at 14% Cr; thus the field of γ -iron is limited to alloys containing less than 14% Cr. The temperature of the A2 transformation rises to a maximum of 771° with 2% Cr, then falls steadily in an almost straight line to 0° with 75% Cr. The heat evolved in the A3 and A4 transformations decreases rapidly with increase in the chromium content of the alloys.

A. R. POWELL.

Annealing effects of certain copper-nickel-aluminium-manganese alloys. L. J. WOOD (J. Physical Chem., 1927, 31, 1693—1703).—The influence of manganese on the ternary system copper-nickel-aluminium has been investigated by a comparison of the densities, corrosion resistance, and microstructure of the sand-cast alloys before and after annealing at 800±10° for 10 days. Alloys of the system copper-nickel-aluminium having the ratio copper/nickel equal, approximately, to 2, and containing 2—6% Al, form, essentially, an α -solid solution when sand-cast and quenched from just below a red heat. Addition of 1% of manganese tends to cause the separation of a second constituent which behaves as an α - β eutectic, and which is regarded as such. Chill-casting minimises the amount of this

constituent in the alloy as also does annealing, which further causes a decrease in density and in corrosion. The maximum combination of valuable properties decreases with an increase in the amount of α - β constituent. The relation between the ratio of copper to nickel and aluminium is also of importance.

L. S. THEOBALD.

Tensile tests on alloy crystals. IV. Copper alloy containing 5% Al. C. F. ELAM (Proc. Roy. Soc., 1927, A116, 694—702; cf. B., 1927, 558).—Crystals of a copper-aluminium alloy containing 5% Al were found to be very much cored, two directions of cores generally being formed irrespective of the orientation of the crystal to the axis of the rod, the directions agreeing in every case with planes parallel to the cube faces. Distortion measurements were made on both annealed and un-annealed crystals, the coring being partially removed by prolonged annealing. The results confirm those obtained with copper-zinc alloys (*loc. cit.*), and fresh evidence is adduced on the effect of non-homogeneity of structure on the distortion. Both slip-plane and direction of slip appear to be uninfluenced by coring, but, as is shown by the effect of annealing, uniformity of structure tends towards greater strength. The copper-aluminium alloy is harder than copper at the beginning of the test, but copper hardens more rapidly than the alloy. The alloying element appears to facilitate slip rather than to impede it.

L. L. BIRCHSHAW.

Removal of iron [from molten metals]. B. BOGITCH (Compt. rend., 1927, 185, 1046—1048).—Iron may readily be removed from copper in a converter without serious loss of the latter, but nickel and cobalt are more readily oxidised, and even when the temperature can be controlled so as to produce warm and fluid slags large quantities may be lost. At high temperatures also the lining of the converter is attacked. In the author's de-ironing process a jet of compressed air strikes a thin stream of molten metal and atomises it into droplets which readily oxidise. These are subsequently collected from the floors and walls of the vessel in the solid state and treated in the electric furnace. Silica is not required at this stage and, since the slag is fluid and consists of almost pure oxides, the losses are reduced to a minimum.

J. GRANT.

Metallographic investigation of metallic material [boiler tube]. F. HANAMAN (Arkiv Hemiju, 1927, 1, 236—242).—Metallographic examination of a burst steel boiler tube shows that it must have been heated at least to 850° and then quenched with steam, thereby causing the formation of martensite; on reheating at 750° it returns to its original homogeneous structure.

R. TRUSZKOWSKI.

Sorting, classification, and briquetting of chrome and manganese ores. K. R. KRISHNASWAMI (J. Indian Inst. Sci., 10A, 65—69).—The quality of the deposits of chrome and manganese ores in Mysore is not distinguished easily without recourse to analysis, but by a systematic examination of a number of samples it was found possible for different persons to agree within 1.5% for each type by inspection of a newly fractured surface. It must not be assumed that samples of similar appearance from different localities will have

the same chromium content. No such correlation between fracture and composition has been found for manganese. Considerable improvement in the quality of some chrome ores was effected with little loss of chromium by washing on a laboratory Wilfley table, but the results were not so satisfactory for manganese ores. Experiments on the feasibility of briquetting powdered ore for bulk transport included the use of tar, clay, lime, magnesite, and magnesium oxychloride as bonds, but without success. Briquettes made with 1.5% of a 35% solution of sodium silicate and heated to 200° were sufficiently strong to withstand rough handling.

C. A. KING.

PATENTS.

Direct production of liquid pig iron and steel from finely-divided materials. K. STRAUSS (G.P. 442,776, 22.7.25).—The finely-divided mixture to be charged to the blast furnace is blown through a series of arcs by means of a current of hot flue gas, whereby it is pre-heated and the ore is partially reduced and sintered.

A. R. POWELL.

Treatment of iron. I. R. VALENTINE, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,648,750, 8.11.27. Appl., 29.12.23).—Iron to be malleabilised is melted in a cupola and oxidised in an electric furnace to reduce its carbon content. After desulphurising the charge, silicon is added and the product cast.

F. G. CROSSE.

Protection of iron from rusting. A. LÉVY (F.P. 617,890, 10.11.25).—After removal of grease the iron is galvanised in the usual way and then immersed for a short time in a solution containing a salt of nickel or copper, so as to produce a thin layer of either metal on the zinc. Finally the article is nickel-plated in any suitable bath. This procedure prevents partial dissolution of the zinc in the electrolytic nickel bath and obviates the necessity of using high current densities in the final plating operation.

A. R. POWELL.

Manufacture of steel. KOHLSWA JERNVERKS AKTIEBOLAG (B.P. 263,811, 23.12.26. Swed., 23.12.25).—The initial hardness and resistance to wear of high-percentage, austenitic, manganese steel may be increased by the addition of about 3.7% each of one or more of the elements, chromium, tungsten, or molybdenum. [Statutory ref. is directed to B.P. 124,817, 18,834 of 1906, and 25,794 of 1903].

M. E. NOTTAGE.

Non-rusting chromium steel. STEIRISCHE GUSSSTAHLWERKE A.-G. (Swiss P. 118,765, 7.9.25. Austr., 24.9.24).—A non-rusting steel, having good machinable properties and suitable for making table cutlery and surgical knives, contains 0.3—0.6% C, 11—15% Cr, 0.4—1% Mo, and 0.2—0.8% Ni.

A. R. POWELL.

Superheating of blast-furnace and cupola-furnace slag and addition of certain lacking constituents. H. BÜHRMANN (B.P. 271,087, 11.5.27. Ger., 15.5.26).—Fluxes, e.g., alkalis or metallic oxides, in powder form, are added to liquid slag from a blast or cupola furnace, and the enriched slag is caused to flow in a thin layer or stream through a furnace, mixing of the constituents being effected by a series of cascades. The enriched slag is useful for building purposes.

C. A. KING.

Refining of copper. E. H. JACOBS, Assr. to ELECTRICAL ENGINEERS EQUIPMENT Co. (U.S.P. 1,648,947,

15.11.27. Appl., 26.3.23).—Copper is melted under a layer of reducing material such as charcoal, and just before pouring about 0.28% by wt. of a purifying flux containing 66.7% Cu, 20.0% of phosphor-copper, 6.7% of copper sulphate, and 3.3% each of fluorspar and borax is added. M. E. NOTTAGE.

Magnetic alloys and their application in the manufacture of telegraphic and telephonic cables. W. S. SMITH and H. J. GARNETT (B.P. 279,549, 27.7.26. Cf. B.P. 224,972; B., 1925, 76).—An alloy consisting of 69–71% Ni, 14–15% Fe, 14–16% Cu, with, if desired, not more than 0.5% Mn and traces of a deoxidiser, has a magnetic permeability which is almost independent of the strength of the field between $H = 0.001$ and $H = 0.1$ gauss. The alloy should be heated at a temperature (880°) above that of the magnetic change point and cooled at a rate (80°/min.), such that its initial permeability is of the order of 6000. M. E. NOTTAGE.

Reduction of zinc ores or other zinc-bearing material. COMP. DES MÉTAUX OVERPELT-LOMMEL (B.P. 279,697, 9.3.27. Belg., 26.1.27).—Plasticity is given to zinc ores by kneading them together with a binder; oxide ores are usually sufficiently plastic with water alone, and iron sulphate or sulphuric acid is a suitable agent for calamines. The kneaded ore is expressed through a die to form threads 2–4 mm. in diameter and 2–15 mm. long, which are dried or roasted as necessary, and afterwards reduced in zinc retorts, the reducing agent preferably being 1–8 mm. in size. C. A. KING.

Treatment of finely-divided oxides [of zinc, tin, or lead] prior to reduction. F. A. GRUESSNER and G. GRUESSNER (G.P. 442,620, 29.11.25).—The material is treated with a solution of an alkali and the crumbly mass so obtained is mixed, while still moist, with a reducing agent. A. R. POWELL.

Magnesium alloy. J. A. GANN, Assr. to DOW CHEMICAL CO. (U.S.P. 1,649,521, 15.11.27. Appl., 27.10.21).—An alloy containing 80–99.5% Mg and 20–0.5% Ni is claimed, having a tensile strength exceeding 18,000 lb./in.², Brinell hardness 45 (approx.), and single-blow impact toughness of 40–30. F. G. CROSSE.

Treatment of aluminium screenings etc. J. G. G. FROST, Assr. to NAT. SMELTING CO. (U.S.P. 1,648,262–3, 8.11.27. Appl., [A] 28.7.24. Renewed 20.8.27; [B] 13.6.25).—(A) Aluminium dross screenings are digested with a solution of sulphur dioxide, and the resultant solution of aluminium compounds is separated from the insoluble material and boiled to precipitate aluminium hydroxide. (B) The aluminium hydroxide is precipitated without removing the material insoluble in sulphurous acid and, after filtration, the residue and precipitate are further treated to dissolve their content of aluminium. A. R. POWELL.

Apparatus for use in refining lead bullion and similar operations. G. K. WILLIAMS (B.P. 267,105, 17.2.27. Austral., 6.3.26).—Lead bullion is treated in deep narrow kettles having a spout outlet from the bottom by which metal may be removed intermittently. The kettle is surrounded by two or more independent heating flues, the upper part being required to be hotter than the lower, and the spout is also heated separately.

A stirring arrangement is fitted in the upper portion of the kettle, and the process is maintained continuous, if desired, by arranging a series of the melting pots stepwise so that the outlet of one discharges into the rest.

C. A. KING.

Manufacture of alloys of lead with the alkaline-earth metals. W. KROLL (G.P. 443,641, 30.12.25).—Lead is heated with the carbide of the alkaline-earth metal in an iron crucible in a vacuum or in an atmosphere of hydrogen or argon. The exclusion of air permits the action to be carried out at relatively low temperatures.

A. R. POWELL.

Bearing metal. S. CHOLEWINSKI (B.P. 272,889, 1.6.27. Poland, 18.6.26).—An alloy suitable for journal bearings and having a Brinell hardness of 29–40 consists of 97–98% Pb, 0.35% Ca, 0.45% Ba, 0.30% Sr, 0.50% Cu, and 0.40% Na. F. G. CROSSE.

Treatment of mercury-bearing ores. R. W. HYDE, Assr. to DWIGHT & LLOYD METALLURGICAL CO. (U.S.P. 1,650,360, 22.11.27. Appl., 10.5.24).—The finely-divided material containing mercury, mixed with just sufficient of a combustible to support combustion in the mixture, is spread in a uniform thin layer over a pervious support. The surface of the mixture is ignited, and combustion proceeds at a uniform rate from top to bottom through it, the temperature being maintained below the fusion point of the ore. The products of combustion are withdrawn and the volatilised mercury is condensed. M. E. NOTTAGE.

[Tarnish-resisting] silver alloys. R. P. JOSEPH and W. F. SCHNORR (B.P. 280,073, 7.2.27).—A standard silver alloy highly resistant to tarnishing comprises 92.5% Ag together with tin, zinc, aluminium, and gold in such proportions that the tin is at least one half of the sum of these four metals, the zinc one quarter, and the gold not more than one tenth, whilst the aluminium content is less than that of the gold. Part or all of the tin and zinc may be replaced by cadmium. A satisfactory alloy contains 92.5% Ag, 4.8% Sn, 2.1% Zn, 0.4% Au, and 0.2% Al. A. R. POWELL.

Recovery of platinum [from residues]. D. C. SMITH (U.S.P. 1,649,786, 15.11.27. Appl., 3.12.23).—Material containing platinum in a soluble form is extracted with water and the solution treated with a metal which liberates hydrogen and forms a salt. The platinum precipitate is collected and purified.

A. R. POWELL.

Preparation of uranium in a coherent mass. H. C. RENTSCHLER and J. W. MARDEN, Assrs. to WESTINGHOUSE LAMP CO. (U.S.P. 1,648,962, 15.11.27. Appl., 22.8.22).—Uranium powder, compressed into a compact mass in a closed vessel, is protected by an atmosphere of inert gas and transferred to a heat-treatment furnace, which is highly evacuated and wherein the uranium is heated at such a rate that occluded gases and volatile impurities are substantially completely expelled without causing the mass to disintegrate. The mass is then heated at a white heat and, finally, at least to its sintering temperature. J. S. G. THOMAS.

Manufacture of thorium alloys. E. C. R. MARKS. From KEMET LABORATORIES, INC. (B.P. 279,274, 17.12.26).—Alloys containing thorium and a refractory metal or

metals of the tungsten group (*e.g.*, tungsten and/or molybdenum) are prepared by heating a compressed mixture of thorium hydride and the metal at a temperature (about 1450°) sufficient to dissociate the thorium hydride and further to sinter the compressed rod. Thorium hydride may be formed by heating thorium in an atmosphere of hydrogen. C. A. KING.

Mineral separator. R. S. BALDWIN and W. A. JOHNSTON (U.S.P. 1,648,333, 8.11.27. Appl., 3.8.25).—A series of precipitating tanks, each having a gold-collecting cup, is combined with a concentrator table which is supported so as to oscillate over a second tank. Means are provided for elevating the material from the first tank to the table, the latter being adapted to retain values and to discharge lighter tailings over the sides of the table to the second tank, also for supplying water to the table, transferring heavier tailings from the table to the third tank, and for disposing of tailings from the third tank. M. E. NOTTAGE.

Ore separator. D. H. HERBERT (U.S.P. 1,648,478, 8.11.27. Appl., 14.11.25).—A shaking table provided with a collecting pocket is combined with a longitudinally inclined, transversely disposed, rotary screen, an inlet hopper being fixed between them. At the rear of the table is a tailings conveyor, whilst between the pocket and the conveyor is a magnetic separator. M. E. NOTTAGE.

Separation of minerals from one another and removal of gangue from flotation froth. T. FRANZ (G.P. 442,642, 5.12.19).—By the addition of alkalis, dyestuffs, oxidising agents, organic compounds, etc. to a flotation froth, one or more constituents thereof are precipitated by electro-capillary forces. The same effect may be produced by the direct action of a suitable electric current. A. R. POWELL.

Flotation process for sulphide minerals. A. W. HAHN and C. M. NOKES (U.S.P. 1,649,685, 15.11.27. Appl., 10.4.25).—In the preferential froth flotation of sulphide minerals the pulp contains small quantities of aluminium sulphate and an alkali cyanide. C. A. KING.

Sintering, desulphurising, or removing volatile constituents from ores and metallurgical products.

A. L. MOND. From METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 279,693, 28.2.27).—In the treatment of ores on a furnace grate as of the Dwight-Lloyd type, the quantity of air supplied to the ignition point is just sufficient to attain the desired high temperature, and then the air supply is increased to a maximum theoretical value in order to concentrate the roasting operation in as short a space as possible, compatible with the permeability and fuel content of the charge. This is accomplished by fitting two or more fans to control the air supply in the different zones of the furnace, the zones possessing a number of suction compartments each under separate control. C. A. KING.

Production of alloys in a finely-divided form. E. SEYFFERTH (G.P. 443,742, 16.7.25).—A mixture of the finely-divided metals is heated in an atmosphere of inert gas or in a vacuum until reaction takes place. Alternatively, a mixture of the powdered oxides is heated in a reducing atmosphere. The powders obtained

by either method are homogeneous throughout and suitable for the manufacture of pressed articles or, after polishing, for decorative purposes.

A. R. POWELL.

Removal of deposits, scale, or incrustations from metal. G. RÜTSCHÉ (B.P. 280,099, 13.4.27. Addn. to B.P. 253,073; B., 1926, 1018).—An incrustated metal surface is subjected to the blast of a large flame produced by burning in an excess of oxygen a mixture of oil and oxygen issuing from a mixing chamber in which it has been preheated before reaching the burner nozzle. Oil is conveyed to the nozzle by means of an asbestos wick, and is volatilised while passing through the mixing chamber. C. A. KING.

Rendering joints, rivets, etc. water- and/or steam-tight. METALLISATION, LTD., and A. A. ARNOLD (B.P. 279,584, 25.8.26).—The cleaned joints etc. are sprayed with a suitable metal such as iron, nickel, or copper, thereby penetrating into and filling up any open spaces between the exposed edges; a very thin film of aluminium followed by a coating of a reducing substance may afterwards be applied and the whole heated (cf. B.P. 259,289; B., 1926, 984). M. E. NOTTAGE.

Electrodeposition of zinc. U. C. TAINTON (B.P. 280,103, 29.4.27).—In the electrolytic recovery of zinc from zinc ores, the impure zinc sulphate solution is electrolysed, using an anode of lead alloyed with 1% of silver and preferably also with arsenic. Manganese dioxide deposited at the anode is improved in quality if the lead anode contains substantial proportions of silver and tin (or arsenic). Power consumption is reduced materially by using two perforated silver-lead plates between each tier of cathodes and spacing the anodes and cathodes close together. C. A. KING.

Electrolytic extraction of pure aluminium from crude aluminium, alloys, etc. ALUMINIUM-IND. A.-G. (B.P. 272,246, 7.6.27. Ger., 7.6.26).—In the electrolytic production of aluminium a solid anode of impure aluminium is used, together with an electrolyte containing a greater molecular concentration of an aluminium halide than of the other metal (alkali) halides present. It is advisable to conduct the operation in a closed vessel. C. A. KING.

Electrolytic production of metals. A. C. JESSUP (B.P. 256,610, 30.7.26. Fr., 5.8.25).—The electrolyte used in the manufacture of magnesium and the alkaline-earth metals is heavier than the metal produced, which forms an upper layer protected by salts of low density. The electrolysis is conducted in a vessel of conducting material which is preferably capable of alloying slightly with the metal produced, the molten metal layer forming the active cathode, and the container the anode. The initial cathode may be a perforated metal plate. C. A. KING.

Fusing of metals by the electric arc process. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of P. P. ALEXANDER (B.P. 259,590, 7.10.26. U.S., 7.10.25. Addn. to B.P. 257,955; B., 1927, 449).—For purposes of arc welding in an atmosphere of a mixture of hydrogen and nitrogen, ammonia is dissociated to form a suitable mixture of gases, or the proportion of nitrogen may be increased by burning air in the dissociated gases and

removing the water vapour formed. Organic ammonium compounds may be used similarly after dissociation if the presence of carbon or carbon gases is not deleterious.

C. A. KING.

XI.—ELECTROTECHNICS.

Nitrogen fixation. COLIN and TARTAR.—See VII.

PATENTS.

Electric induction furnace. ELECTRIC FURNACE CO., LTD. From E. F. NORTHRUP (B.P. 279,733, 21.5.27).—In an electric induction furnace comprising a plane loop duct or ducts communicating at the ends with the pool, one cross-sectional dimension of the duct or ducts being greater than the other, the greater cross-sectional dimension or the plane of the duct or ducts is arranged obliquely to the axis of a cylindrical transformer coil, so that a non-uniform inductive action, producing circulation, is obtained.

J. S. G. THOMAS.

Evacuated [electron-discharge] device and method of exhaust. R. E. MYERS, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,648,958, 15.11.27. Appl. 3.5.23).—An electron-emitting cathode, coated with the carbonates of alkaline-earth metals and sealed into an exhausted vessel, is heated so that oxides of carbon are liberated from the carbonates. The oxides of carbon are ionised by passage of an electric discharge and subsequently removed, and a high vacuum is produced in the envelope by a metallic clean-up agent.

J. S. G. THOMAS.

Metallic filaments for electric incandescence lamps. R. STEWART, Assee. of NEUE GLÜHLAMPEN G.M.B.H. (B.P. 277,634, 11.8.27. Ger., 18.9.26).—Wires, having a single-crystal or long-crystal structure, are bent in the cold state to an arc of internal radius approximately equal to the diameter of the wires, whereby the grid-structure of the crystal is partially destroyed.

J. S. G. THOMAS.

Manufacture of oxide-coated cathodes. W. B. GERO and G. W. HALLOCK, Assrs. to WESTINGHOUSE LAMP CO. (U.S.P. 1,648,941, 15.11.27. Appl. 16.7.26).—A coating composition for the cathodes consists of a suspension of alkaline-earth carbonates in a solution of potassium permanganate.

M. E. NOTTAGE.

Preparation of electric accumulator plates. EDISON SWAN ELECTRIC CO., LTD., and G. E. WEBSTER (B.P. 279,926, 8.7.26).—Electric accumulator lead plates are "formed" in a solution of sulphuric acid containing 1–3% by vol. of perchloric acid, and are then dried at about 116°.

J. S. G. THOMAS.

Negative electrode for storage batteries. R. C. BENNER and L. C. WERKING, Assrs. to PREST-O-LITE STORAGE BATTERY CORP. (U.S.P. 1,649,280, 15.11.27. Appl. 10.9.23).—A storage battery comprises a negative electrode consisting of a mixture of active material with 2–10% of barium sulphate and 0.35–5% of lampblack, and a separator pervious to electrolyte but impervious to the active material, held in direct contact with the active material to counteract the disintegrating action of the barium sulphate and lampblack.

J. S. G. THOMAS.

Apparatus for electro-deposition. INTERNAT. COPPERCLAD CO., Assees. of E. M. WANAMAKER (B.P. 276,306,

28.5.27. U.S., 19.8.26).—In apparatus for electro-deposition, more especially for use in the manufacture of "copper-clad" roofing elements, as described in E.P. 238,230, 255,113—4 (B., 1927, 81, 144, 47), positive and negative bus-bars are arranged near the electrolytic cell, and a frame which is movable along the cell is provided with shoes, one of which makes contact with the positive bars and is electrically connected to all the anodes, whilst the other makes contact with the negative bus-bar and is electrically connected to all the cathodes.

J. S. G. THOMAS.

Electroplating apparatus. J. G. NEWBY and C. B. JERRED (B.P. 279,616, 9.10.26).—A closed rotatable barrel of insulating material, adapted to receive the electrolyte and articles to be plated, has anode plates mounted in recesses behind perforated celluloid or other plates in or upon its end walls, and cathode contact studs or plates with which the articles to be plated make contact, arranged at intervals along its periphery.

J. S. G. THOMAS.

Electrical [gas] precipitator. W. A. SCHMIDT, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,650,097, 22.11.27. Appl. 9.9.25).—In a device for separating suspended particles from gases, a high-resistance collecting electrode and a discharge electrode, insulated therefrom and consisting of conducting material covered with material facilitating uniform electrical discharge, are maintained at a high difference of potential.

J. S. G. THOMAS.

Electrical gas-purifying plant. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of R. HEINRICH (G.P. 442,876, 18.4.24).—The dust-collecting chamber is arranged below the electrodes and the whole of its cross-sectional area is covered with a permeable sheet provided with undulations disposed transversely or inclined to the gas stream, so that passage of gas to the dust-collecting chamber is prevented. The sheet can be used as an auxiliary precipitating electrode, and the lower edges of the discharge electrodes are provided with special discharge devices.

J. S. G. THOMAS.

Apparatus [discharge electrode] for electrical separation of suspended particles from gases. E. ANDERSON, Assr. to INTERNAT. PRECIPITATION CO. (U.S.P. 1,650,105, 22.11.27. Appl. 17.9.25).—A discharge electrode comprises a conducting member surrounded by a continuous coating of semi-conducting material.

J. S. G. THOMAS.

Working of electrical gas purifiers. METALLBANK U. METALLURGISCHE GES. A.-G., Assees. of E. HUETER (G.P. 443,019, 13.12.25).—The deposit of dust on the electrodes, and more especially that on the precipitating electrode, is removed by a short interruption of the direct voltage operating the plant and the establishment of a lower alternating voltage during that interval. Two or more discharge electrodes and purifiers can be arranged in the gas stream so that removal of dust from the electrodes can be effected alternately without the necessity for interrupting or reversing the gas stream.

J. S. G. THOMAS.

Photo-electric cell. W. S. SMITH and N. W. McLACHLAN (B.P. 279,937, 3.8.26 and 12.3.27).

Gas-filled photo-electric cell. GEN. ELECTRIC CO., LTD., and N. R. CAMPBELL (B.P. 280,077, 15.2.27).

Manufacture of electrical resistance bodies and machine therefor. S. LOEWE (B.P. 256,264, 31.7.26. Ger., 1.8.25).

Coke for electrodes (B.P. 269,849).—See II.

Magnetic alloys (B.P. 279,549).—See X.

XII.—FATS; OILS; WAXES.

Vulcanisation of oils. P. STAMBERGER (Rec. trav. chim., 1927, 46, 837—840).—When a mixture of rape oil with 20% of sulphur is heated at 170—180° an exothermic reaction occurs during which hydrogen sulphide is evolved. The viscous residue, after extraction with acetone and then with benzene, leaves an elastic solid which is insoluble in organic solvents. All three fractions have approximately the same elementary composition—64.33% C, 9.7% H, 16.38% S; the acetone-soluble portion (mol. wt. 990—1084) is a viscous liquid, the benzene-soluble portion (mol. wt. 2130—2285) has a honey-like consistency. The fractions on hydrolysis yield acids having mol. wt. of 807—831, 854—864, and 891—831 respectively, and approximately the same average composition (65.91% C, 9.82% H, 13.7% S). The figures indicate that a small amount of the total sulphur is combined with the alcoholic residue of the water. G. A. C. GOUGH.

Lower fatty acids of coconut oil. E. R. TAYLOR and H. T. CLARKE (J. Amer. Chem. Soc., 1927, 49, 2829—2831).—Fractionation of 130 kg. of methyl esters of coconut oil acids indicates that this oil contains lower fatty acids as follows: hexoic (0.46%), octoic (8.7%), deoic (5.6%), lauric (45.0%), and myristic acids (16.5—18%). F. G. WILLSON.

Determination of the iodine value [of oils] in aqueous emulsions. B. M. MARGOSCHES and K. FUCHS (Z. anal. Chem., 1927, 72, 185—187).—Repetition of the work of Fialkov (B., 1927, 304) showed that very low figures were obtained for the iodine value of oils by following the prescribed directions; good results were obtained only by a very much longer time of contact than 5 min., the time varying with different oils.

A. R. POWELL.

Adhesion of waxes. McBAIN and LEE.—See I.

Refractometer for oils. LöWE.—See I.

Jute seeds. SEN.—See XX.

PATENTS.

Extraction of fats. H. V. ATWELL (U.S.P. 1,648,102, 8.11.27. Appl., 27.2.23).—The powdered, fat-containing material of vegetable origin is moistened with water, insufficient to form a paste, before treatment with a solvent. H. ROYAL-DAWSON.

Soaps containing succinic acid (B.P. 279,575).—See XX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Causes of livering in paints, enamels, and printing inks. P. E. MARLING (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1927, Circ. No. 319, 535—540).—Mixtures of zinc oxide and variously treated linseed oils

(e.g., blown, heat-treated, free fatty acids added) were observed for livering. It is shown that the acid value of an oil is not a deciding factor in this connexion, its previous history being more significant. The formation of metallic soaps that later redissolve is discussed.

S. S. WOOLF.

Zinc oxide in exterior mixed paints. E. H. BUNCE (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1927, Circ. No. 319, 541—550).—The advantages of using zinc oxide in paints for exterior use are:—increased gloss of finished surface, protection of binding material from actinic rays, hardening of the film, and neutralisation of acidic decomposition products. Each of these points is amplified.

S. S. WOOLF.

Kauri reduction test as applied to flat wall paints. R. H. EVERETT (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1927, Circ. No. 319, 570—577).—The kauri reduction test does not give concordant results in the hands of various operators when applied to flat wall paints, nor are the results substantiated by exposure tests on the paints. Reliable conclusions as to the life of this type of paint on exposure may be drawn from the composition by volume of the paint. S. S. WOOLF.

Evaluation of the kauri-butyl alcohol solvency test [for varnish thinners]. S. R. KIEHL (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1927, Circ. No. 319, 585—596).—A "solvency" test for varnish thinners dependent on their degree of miscibility with a standard solution of kauri in butyl alcohol is described in full detail. The applications of this test to the specification of individual thinners and to the examination of binary mixtures are illustrated. It is shown that mixtures of equal "solvency" may exercise similar functions when used as thinners. S. S. WOOLF.

Pigments for iron and steel primers. E. W. FASIG and J. M. PURDY (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1927, Circ. No. 319, 611—622).—A general dissertation on rusting and rust prevention is followed by details of tests on a number of pigments when placed in wet contact with steel. The results of the tests, which were still proceeding, indicated that among the foremost rust inhibitors, litharge compares favourably with zinc chromate, and it is suggested that red lead pigments with as much as 10% of free litharge are preferable to pure red lead in this connexion. S. S. WOOLF.

Causes of instability of varnishes on standing. J. F. MAGUIRE (Amer. Paint & Varnish Manufs.' Assoc., Nov., 1927, Circ. No. 319, 564—569).—A brief summary of the effects of the constituents of varnishes, mixing, ageing, chilling, and centrifuging on the stability of colloidal varnish systems. S. S. WOOLF.

Polish turpentine. J. FLATAU and A. KORCZYŃSKI (Rocz. Chem., 1927, 7, 246—260).—Turpentine originating from Eastern and Western Poland has been examined. Treatment of the fractions of the former of b.p. 160—168° with hydrochloric acid yields a considerable quantity of bornyl chloride, which is not given by Polish Pomeranian turpentine. In the latter case only terpin hydrate is obtained by the action of dilute acids and subsequent hydrolysis. R. TRUSZKOWSKI.

Adhesion of resins. McBAIN and LEE.—See I.

Stability of varnishes and lacquers. BARTELL and OSTERHOF.—See I.

PATENTS.

Printing in greasy inks with gelatin printing surfaces. SANDOR G.M.B.H. (B.P. 263,125, 6.12.26. Ger., 18.12.25).—To avoid the formation of undesirable reliefs in the process of printing in greasy inks with gelatin surfaces, suitable substances are added to the wetting medium with which the gelatin is treated, in order to reduce or delay the swelling of the gelatin. In addition, the temperature of the medium is raised above 22°, the taking up of greasy ink by the high-lights being thereby prevented. S. S. WOOLF.

Printers' ink. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 270,671, 13.4.27. Ger., 5.5.26. Addn. to B.P. 239,113; B., 1925, 891).—Manganese dioxide of low bulk density (0.9 or less) is prepared by precipitating a manganese salt with excess of sodium carbonate solution, passing in chlorine until the brownish-black precipitate has completely separated, and drying this precipitate to a water content not exceeding 1.5%. The use of such light-weight manganese dioxide in printers' ink obviates clogging of the type in rotary printing machines. S. S. WOOLF.

Obtaining indelible writings and prints. U. A. D'INZEO (B.P. 280,088, 9.3.27).—Paper or other suitable material, sensitised with a liquid containing gallic acid or tannic acid, is written or printed on with a solution of ferrous nitrate or a chromate, *e.g.*, potassium chromate, in water, alcohol, etc. S. S. WOOLF.

Production of colour bases for varnishes etc. J. S. WILSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 278,765, 12.4.26).—An aqueous vat dye paste is mixed with a suitable medium (castor oil, linseed oil, turpentine, etc.) and the water is evaporated, preferably in a vacuum at 50–60°. C. HOLLINS.

Treatment of decamphorated oil of turpentine. I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (B.P. 255,896, 23.7.26. Ger., 25.7.25. Addn. to B.P. 239,878; B., 1925, 999).—The residue obtained when the non-resinifying portion boiling up to 200° is removed by distillation from decamphorated oil of temperature (*cf.* prior patent) is treated with an oxidising agent, the product being used as a linseed oil substitute. S. S. WOOLF.

Manufacture of titanite oxide. N. SPECIT (U.S.P. 1,649,496, 15.11.27. Appl., 19.5.25. Ger., 24.5.24).—See B.P. 234,518; B., 1925, 670.

Plant for manufacture of paints, enamels, inks, etc. J. R. TORRANCE, and TORRANCE & SONS, LTD. (B.P. 280,041, 30.11.26).

Iron oxide pigment (B.P. 279,283).—See III.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

PATENTS.

Manufacture of elastic, waterproof coatings. H. QUITTNER (B.P. 263,849, 29.12.26. Austr., 30.12.25).—See Austr. P. 104,398; B., 1927, 420.

Method and means for drying rubber or articles containing rubber. TOMLINSONS (ROCHDALE) LTD., and E. W. SMITH (B.P. 280,016, 14.10.26).

XV.—LEATHER; GLUE.

Chrome tanning. V. Properties of differently prepared chromium chloride solutions. E. STIASNY and O. GRIMM (Collegium, 1927, 505–531).—The p_H value of various solutions of normal and 33% basic chromium chloride was determined before and after heating, after ageing for various periods up to three months, after adding a calculated quantity of hydrochloric acid, and, in the case of the basic chloride, after heating both before and after rendering basic respectively. Precipitation values and diffusibility were also determined on the different liquors. The results show that the composition and properties of a solution of chromium chloride depend largely on whether the liquor was heated before being rendered basic, the degree of basicity, on heating or ageing after it was rendered basic, and on ageing after the heating. The effects are indicated by the p_H value of the liquors and the size of the molecules of the basic salts, which determine the colloidal character of the liquor. The following changes are effected by the above treatments:—hydrolysis with the formation of hydroxy-salts or basic salts and free acid; the conversion of hydroxy-salts into "ol" compounds; the further conversion of "ol" bridges into oxygen bridges; the entrance of acid radicals into, or their removal from, the chromium complex; neutral salt effects on the free acid present. When a basic chromium chloride solution was boiled for 5 min. the p_H value altered and only recovered after three days. It also decreased on ageing until it reached 2.79 after four weeks; addition of hydrochloric acid postponed the "ageing" effect. Heating before rendering the solution basic caused chlorine to enter the chromium complex, but there was no chlorine in the complex after the solution had been rendered basic and aged for a short time. Boiling the solution before rendering it basic encouraged the formation of "ol" compounds; "ageing" had the same effect. Boiling was more effective in the formation of "ol" compounds and oxygen bridges after the liquor had been made basic than before. Heating increased the precipitation value both of normal and basic chromium chloride solutions, owing to the formation of "ol" compounds which are less easily precipitable than the hydroxy-compounds. The heating effect was more pronounced with the more basic salts. Ageing diminished the precipitation value of heated solutions, of solutions of normal chloride which had not been heated, and of basic solutions which had been heated before and after rendering basic, but increased the value for basic solutions which had been boiled before rendering basic only. The dialytic properties of the different types of chromium chloride solutions were almost the same, showing that conversion into "ol" compounds does not result in the formation of large molecules unless the basicity exceeds 33%. D. WOODROFFE.

Egg albumin and egg yolk as emulsifying agents in fat-liquoring [of leather]. J. A. WILSON (J. Amer. Leather Chem. Assoc., 1927, 22, 559–565).—A mixture of equal parts of neatsfoot oil and sulphonated neatsfoot oil was mixed with different amounts of fresh whole egg, yolk alone, egg white, and thickened whole egg, respectively, emulsified with water, and kept. The

stability of the various emulsions was determined and plotted as a function of the added egg product. The results showed that thickened whole egg increased the stability much more than fresh egg, and that the yolk alone had very little stabilising power. Egg-white was more stabilising than whole egg. Photomicrographs of leathers treated with various fat-liquors showed that penetration depends directly on the stability of the liquors. Completely penetrated leather was loose. It is necessary in fat-liquoring to lubricate the fibres in the outer layers only, to give strength, softness, and pliability, but an unlubricated layer should be left sufficiently thick to give the leather the necessary tightness and temper. The best fat-liquor distributes the oil in the outer layers of the leather, with the amount decreasing to zero at the middle of the skin. Egg products assist in producing this effect.

D. WOODROFFE.

Influence of neutral salts on the plumping of hides. R. O. PAGE and A. W. PAGE (Ind. Eng. Chem., 1927, 19, 1264—1268).—Pieces of bated cowhide were immersed in solutions of calcium chloride, sodium sulphate, and sodium chloride, of different strengths (0.25—4*N*) and having p_H values of 2, 5, 8, and 11, respectively. The plumping effects on the skins were determined by measuring the increase in thickness with a thickness gauge. The p_H value of the solutions greatly affected the influence of the neutral salts added. In acid solutions (p_H 2) the addition of each of the salts reduced the plumping to a minimum of 0.8 times the original thickness with 0.75*N*-solutions of neutral salt. At greater concentrations the plumping increased again, the increase being greatest with the calcium chloride. At p_H 5 and 8 sodium sulphate gave a flat plumping curve with a minimum at 0.7*N*. Sodium chloride also gave a maximum at about this concentration, but it was less pronounced at p_H 8 than at p_H 5. Increased concentration of calcium chloride increased the plumping at both these p_H values, rapidly at first, then more slowly for *N*- to 3*N*-solutions, and finally more rapidly up to 4*N*. The plumping was greater at p_H 8 than at p_H 5. Hide pieces plumped in the more concentrated solutions, lost calcium chloride rapidly and completely on washing, but lost their plumping much more slowly. Hide plumped in 4*N*-calcium chloride retained its plumping even after soaking for one week in distilled water. At p_H 11, increased concentration of sodium chloride depressed the plumping, but much less than in acid solution. The least plumping (1.27) was obtained at the maximum concentration studied. Sodium sulphate depressed the plumping much more rapidly to a minimum 0.84 at 1.5*N*, and then there was a slight increase in stronger solutions. Hide plumped in sodium hydroxide solutions of p_H 12.5 reached apparent equilibrium after one week, but calcium hydroxide solutions of the same p_H value required two weeks. In neither case did the hide fall to its original thickness on restoring it to its original p_H value, but bating helped to reduce it. The nitrogen dissolved from the hide by solutions of neutral salts increased as the plumping in such solutions increased, from which it appeared that these two effects were due to the same cause. Hydrochloric acid was used to give acid solutions for the chlorides and sulphuric acid for the sulphates.

Both acids were tried with sodium chloride, but there was no difference in the influence of the salt on the plumping.

D. WOODROFFE.

Swelling of hide. H. OWRUTSKY (Collegium, 1927, 531—534).—Pieces of un haired, fleshed hide were swollen in 0.1*N*-solutions of hydrochloric and sulphuric acids, and sodium hydroxide, respectively, for 24 hrs., drained, and dried at 24—25°. Four weighings were made during the drying period and curves drawn to show the rate of drying. The results show that acid swelling causes a greater absorption of water than alkaline swelling, and monobasic acids more than dibasic. The acid-swollen pelt lost more water in drying than that swollen by alkalis. The alkaline drying curve was steeper than the acid curves, and the sulphuric acid curve than the hydrochloric acid curve. The water combined with alkaline-swollen pelt is in a weaker state of combination than that in acid-swollen pelt. These results confirm those of Loeb, Procter, and Wilson.

D. WOODROFFE.

PATENTS.

Tanning of animal hides. I. G. FARBENIND. A.-G., Assees. of K. H. MEYER and H. SCHÜTTE (G.P. 441,769, 15.11.23. Addn. to G.P. 420,646; B., 1926, 455).—The products obtained by the action of concentrated sulphuric acid or its monohydrate on lignite tar or low-temperature tar, or fractions derived from these, are used instead of the products described previously (*loc. cit.*).

L. A. COLES.

Tanning of animal hides. J. SCHÄFER, Assr. to J. R. GEIGY, Soc. ANON. (U.S.P. 1,650,541, 22.11.27 Appl., 1.9.26. Ger., 5.8.25).—The hides are treated with a salt of hydrofluosilicic acid before being tanned.

H. ROYAL-DAWSON.

Tanning and dyeing of furs and leather. V. ZETTLITZ, Assr. to A. PFEIFER (U.S.P. 1,649,502, 15.11.27. Appl., 13.1.27. Ger. and Czechoslov., 30.11.26).—Furs or hides are tanned with loam and sodium chloride, then with sodium chloride and alum, then with chrome alum, and finally dyed hot.

B. FULLMAN.

Production of tanning agents from sulphite-cellulose waste liquor. I. G. FARBENIND. A.-G., Assees. of H. SCHÜTTE (G.P. 441,770, 4.4.25).—The liquor, after concentration or evaporation to dryness, is heated with sulphur in the presence of alkaline solutions under such conditions that the product contains little or no insoluble matter. *E.g.*, a mixture of concentrated liquor containing 25% of water, with sulphur and aqueous sodium hydroxide solution, is heated at 110—115° with stirring under a reflux condenser until evolution of hydrogen sulphide slackens, when the product is diluted, filtered if necessary, neutralised, and evaporated to dryness.

L. A. COLES.

Manufacture of leather oil. STANDARD DEVELOPMENT Co. (B.P. 255,908, 27.7.26. U.S., 27.7.25).—A mineral oil (90 pts.) of lubricating grade and having a Saybolt viscosity of 100—300 sec. at 37.5°, is mixed with not less than 5 pts. (8.5 pts.) of sodium sulphonate residue from the acid-treatment of petroleum and 1.5 pts. of cod-oil soap, saponified organic material, or fatty material, and the mixture is heated, cooled, and allowed to settle.

D. WOODROFFE.

XVI.—AGRICULTURE.

Biochemistry of water-logged soils. I. Effect of water-logging on the different forms of nitrogen, on the reaction, on the gaseous relationships, and on the bacterial flora. II. Presence of a deaminase in water-logged soils and its rôle in the production of ammonia. V. SUBRAHMANYAN (J. Agric. Sci., 1927, 17, 429—448, 449—467).—I. Laboratory experiments on the effects of water-logging on soils from Rothamsted and from India are reported. The soils were air-dried, freed from undecomposed plant residues, shaken well with water (100 pts. of soil in 250 pts. of water), and incubated for 40 days, samples being taken for examination at 3-day intervals. There was a marked increase in the content of free and saline ammonia, especially during the first few days, and a slight, though significant, diminution of nitrates. No loss of total nitrogen was observed. The increase in ammonia was correlated with an increase in p_H value. There was no appreciable production of carbon dioxide, but there was diffusion of dissolved oxygen from the surface water into the soil. The results of bacterial counts on several media, together with the lack of production of carbon dioxide, indicate that the formation of ammonia in water-logged soils is not due to biological action. Enzyme action is suggested.

II.—Evidence is given for the occurrence of a deaminase in water-logged soils. The ammonia formed was present mostly in the soil sediment; on allowing the soils to dry, nitrification proceeded rapidly. The production of ammonia took place in presence of volatile antiseptics. An active preparation of the enzyme was extracted from the soil, and the enzymes from cultures of the mixed microflora of the soils also showed deaminising action. Only simple amino-compounds (glycine, aspartic acid, asparagine) were acted upon by the deaminase.

C. T. GIMMINGHAM.

Physiological behaviour of moist, air-dried, and repeatedly dried soils. V. KAŠ (Sborn. Czechoslov. Akad. Zemedel., 1926, 1, 89—152).—The changes in physical, chemical, and biological properties of soil after drying, alternate wetting and drying, and freezing are described. The differences are considered to be a direct indication of productivity. CHEMICAL ABSTRACTS.

Acidity, degree of saturation, and lime requirement of soils on the basis of pot experiments. P. OBST, K. WODARZ, and D. MEYER (Z. Pflanz. Düng., 1927, 10A, 65—90).—Comparison is made of the effects of lime, precipitated chalk, and potassium carbonate in reducing soil acidity. Precipitated chalk was the most rapidly effective and potassium carbonate the least. The "exchange" acidity was neutralised within four weeks in most cases, but the calculated amount of lime necessary for this proved insufficient to remove all acidity. The lime requirement of various soils for different crops showed considerable differences in the degree of acidity necessary for optimum growth. In a few cases the addition of more than the optimum quantity of lime reduced crop yields. The physiological reaction of nitrogenous fertilisers is shown to be of importance in acid soils. The "degree of saturation" characteristic of a neutralised soil differs for soils of

different types, and is not a satisfactory basis for determination of their lime requirement. A. G. POLLARD.

Effect of different manures on soil of strongly acid reaction. A field experiment. E. MÖLLER-ARNOLD and E. FEICHTINGER (Z. Pflanz. Düng., 1927, 6B, 497—501; cf. B., 1926, 959).—In continuation of earlier work, the results of field experiments with various manures on the yield of rye on a very acid soil (p_H 4.0) are discussed. The effect of liming in the spring showed itself in the same year. Sodium nitrate gave a greater increase of yield than ammonium sulphate. C. T. GIMMINGHAM.

Action of phosphoric acid on lime in the presence of clay and pulverulent materials. R. DUBRISAY and (Mlle.) F. DESBROUSSES (Compt. rend., 1927, 185, 1036—1038; cf. A., 1927, 827).—The fixation of phosphoric acid in the soil (*i.e.*, its reaction with calcium carbonate to form an insoluble phosphate) is shown to be inhibited by the presence of absorbing material, particularly clay, even when the proportion of lime is high enough to convert all the acid. The phenomenon is due to the increased solubility of calcium phosphate in water containing carbon dioxide. J. GRANT.

Neubauer's seedling method [for determination of available phosphorus and potassium in soils]. E. GÜNTHER (Z. Pflanz. Düng., 1927, 6B, 502—506).—In using the Neubauer method it is essential to select the rye seed to be used so that the weight per 100 seeds is constant within narrow limits. Experiments indicate that the upper layers of the soil samples tested are not completely exhausted of available phosphorus and potassium by the growth of one lot of seedlings. The weakest point in the method is the difficulty of fixing limiting values for available phosphorus and potassium which will apply to all soils. C. T. GIMMINGHAM.

Evaluation of phosphatic fertilisers by means of their solubility in nitric and citric acids. K. BAMBERG (Z. Pflanz. Düng., 1927, 10A, 100—103).—The effect of the reaction of extracting acids on the solubility of fertiliser phosphates is recorded. The p_H value of citric extracts increases with the period of contact with the fertiliser to an extent characteristic for each fertiliser, and the final value differs for each. The solubility of the phosphate also increases with the contact period, but not proportionally to the p_H value. Variations in p_H values when nitric acid is used differ in nature and extent from those of citric acid. Nitric acid extracts more phosphate from fertilisers than citric acid when used at similar relative concentration. The possibility of the activity of nitrifying organisms affecting the availability of phosphates in soil is indicated.

A. G. POLLARD.

Influence of prolonged cultivation on the nitrogen content of unmanured soils. J. DUMONT (Compt. rend., 1927, 185, 605—608).—The nitrogen content of a piece of unmanured land fell from 2.04 to 1.19 g./kg. during 53 years of cultivation. The annual losses vary considerably, and are much less during the years in which leguminous crops were grown. The loss of nitrogen of another piece of unmanured land, which was cultivated annually for 35 years, was found to be slightly more than

half that of a similar piece of land not regularly cultivated.
G. A. C. GOUGH.

Soil acidity. S. ODÉN (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1927, B, 10—29).—The action of acids on clay consists of the neutralisation of hydroxyl ions on the surface of the particles, together with the aggregation of particles, whereby part of the surface becomes protected from the action of the acid, and finally direct dissolution of mineral particles. The rate and extent of these three processes are largely dependent on the hydrogen-ion concentration of the fluid and on the physical conditions of the mixture. With high hydrogen-ion concentration the coagulating effect predominates, and at low concentrations neutralisation is the main effect. The complete neutralisation of hydroxyl ions is not instantaneous, and this is assumed to be due to the plaited or spongy nature of the clay particle surface and to the consequent lapse of time required for the diffusion of neutralising ions to the interior.

A. G. POLLARD.

Behaviour of acid and alkaline soils with iodine manuring. T. VON FELLEBERG (Biochem. Z., 1927, 188, 326—338).—Animal charcoal adsorbs elementary and ionised iodine much better than do kieselguhr, kaolin, or precipitated calcium carbonate, and these latter substances adsorb elementary iodine better than ionised iodine. The power to set iodine free from added potassium iodide of two alkaline and two acid soils is investigated. With a particular soil, formation of iodine increased with increasing amounts of added iodide, and from 5% of potassium iodide upwards became slower and quite regular. Two acid soils set free considerable amounts of iodine, but only traces from two alkaline soils, the amount of iodine set free varying directly with the acidity of the soil. By mixing the acid soil with 10% of stable manure, its action was almost completely inhibited. With acid soil and iodine manuring, the roots were completely rotted. In the corresponding experiments without iodine, and in similar experiments with the addition of stable manure, the roots were uninjured, and the conclusion is drawn that iodine manuring with acid soils impairs resistance to disease. With four types of soil a comparison is made of the amount of iodine used as manure and the amount absorbed by plants. Increase of iodine affects the leaves more than the roots.

P. W. CLUTTERBUCK.

New form of quinhydrone cell for measurements of hydrogen-ion concentration in soils. L. SMOLIK (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1927, B, 149—150).—The use of the agar bridge and the connecting vessel for potassium chloride solution is obviated, and the whole is enclosed in one vessel.

A. G. POLLARD.

Potentiometric p_H determinations in soil. F. TERLIKOWSKI (Trans. 2nd Comm. Internat. Soc. Soil Sci., 1927, B, 151—155).—Variations in p_H values due to drying of soil samples or alterations in the soil: water ratio of the suspensions follow no definite rule. Changes in p_H with varying soil: water ratio are greatest between the ratios 2:1 and 1:1. Fresh soil samples are recommended for the determination. For the most satisfactory results a soil: water ratio of 1:1 or 1:0.5

should be used, as under these conditions the fluid is well-buffered, and not seriously affected by carbon dioxide.
A. G. POLLARD.

Rapid method for the mechanical analysis of soils. G. J. BOUYOUCOS (Science, 1927, 65, 549—551).—Since most of the physical and physico-chemical properties of soil reside in the colloidal portion, a determination of this fraction by the rapid hydrometer method may replace the tedious mechanical analysis.

A. A. ELDRIDGE.

Mechanical analysis of soils. II. M. KÖHN (Z. Pflanz. Düng., 1927, 10A, 91—99).—Mathematical formulæ underlying standard methods of mechanical analysis do not commonly include temperature effects. The effect of temperature changes on the density and viscosity of water, and therefore on the rate of sedimentation of soil particles, is discussed. Particles of more than 0.2 mm. mean diameter are not seriously affected by changes of temperature, but with finer particles the velocity of settling at 30° may be as much as double that at 5°. Means for calculating a temperature correction are indicated.

A. G. POLLARD.

Micro-determination of nitrogen in soils. M. TANAKA (Bull. Agric. Chem. Soc. [Japan], 1927, 2, 161—162).—An application of Dubsky's method (A., 1919, ii, 169), whereby 0.01 mg. of total nitrogen may be determined in soil is described; the error does not exceed 1%.

CHEMICAL ABSTRACTS.

Determination of nickel in soils. G. SCHIECKENTHAL (Z. Pflanz. Düng., 1927, 10A, 104—107).—Methods for the determination of nickel in soil are described, and many determinations of iron and nickel are recorded. The relative proportions of iron and nickel in soils of similar origin are generally similar. The nickel content of soils of more recent origin is relatively greater than that of older soils.

A. G. POLLARD.

Applicability of Trénel's "acidimeter." G. LAUFER (Z. Pflanz. Düng., 1927, 6B, 486—489).—The use of Trénel's "acidimeter" for electrometric determination of the p_H value of phosphate solutions, soil suspensions, and soil filtrates has been investigated. The method is rapid, and the results are accurate if the standard solution and the unknown solution differ only slightly in p_H value. If solutions differing considerably are compared a much longer time must be allowed before taking the reading, and the results, even then, are not entirely reliable. C. T. GIMMINGHAM.

Crop variation. IV. Experimental determination of the value of top dressings with cereals. T. EDEN and R. A. FISHER (J. Agric. Sci., 1927, 17, 548—562).—A contribution to field experimental technique. The experiment described was designed to compare the effect on the yield of autumn-sown oats of single and double dressings of ammonium sulphate and chloride (equivalent amounts of nitrogen), applied at two different dates (March 15 and June 5). Ninety-six plots were laid out (eight-fold replication of treatments), and the arrangement of the plots and the choice of combination of treatments were such that the effects of soil heterogeneity were eliminated, and each plot yield contributed to the accuracy of all the comparisons desired. A high

degree of accuracy in the results was thus obtained, the standard error for grain being 1.39% and for straw 2.22%. The results showed that a significant response in the yield of grain was confined to the quantity of nitrogenous top-dressing, the effects of kind and date of application being inappreciable, even with the low standard error attained. In regard to yield of straw, the advantage shown by the double dressing was confined to the early application; no differences as between the effects of sulphate and chloride were found.

C. T. GIMINGHAM.

Nutritive and manurial values of sugar beet tops.

H. E. WOODMAN and J. W. BEE (J. Agric. Sci., 1927, 17, 477—488).—The results of digestibility trials on sheep with English-grown sugar beet tops are recorded. The figures for the digestion coefficients of the various constituents are in good agreement with corresponding figures given by Kellner, and it is concluded that sugar beet tops form a palatable feeding stuff of high digestibility, the carbohydrate constituent, consisting partly of sugar derived from the crowns of the roots, being especially digestible. The digestibility of the fibre is also notably high. A study of the lime balances in the sheep during the trials indicate that the lime in the tops may not be in an available form, possibly owing to the presence of oxalic acid in the leaves. Analytical data show that sugar beet tops are extremely rich in inorganic constituents, though the percentage of phosphoric acid is relatively low. Average figures are given from which the manurial value may be calculated.

C. T. GIMINGHAM.

Solubility of phosphorites in salt solutions saturated with carbon dioxide.

K. FLEROW (Z. Pflanz. Düng., 1927, 10A, 109—114).—In the presence of carbon dioxide, comparison of the effect of alkali salts on the solubility of phosphorites indicates that the influence of anions is in the order $\text{SO}_4 > \text{NO}_3 > \text{Cl}$. Increased concentration of salts increases the phosphate solubility, though not in direct proportion. Among the cations calcium is the least effective on the solubility of phosphate. Fertiliser salts affect the solubility of soil phosphates, calcium usually decreasing this.

A. G. POLLARD.

Reactions of Moravian soils. J. SMOLÍK (Vestn. Czechosloven. Akad. Zemedelske, 1926, 219 pp.).—Values of the p_{H} of various types of Moravian and Central European soils are recorded. The quinhydrone method of determination is preferred. CHEMICAL ABSTRACTS.

Laterite and lateritic soils in Sierra Leone. F. J. MARTIN and H. C. DOYNE (J. Agric. Sci., 1927, 17, 530—547).—A study of the red soils of Sierra Leone leads to the suggestion that the classification of laterite and lateritic soils should be based on an examination of the clay fraction. It is proposed that soils in which the silica-alumina ratio in the clay fraction falls below 2.0 should be described as lateritic, and those in which this ratio falls below 1.33 as laterite soils.

C. T. GIMINGHAM.

Contact insecticides. V. Toxicity of amines and N-heterocyclic compounds to *Aphis rumicis*, L. VI. Insecticidal action of the fatty acids, their methyl esters, and sodium and ammonium salts. F. TATTERSFIELD and C. T. GIMINGHAM (Ann. Applied

Biol., 1927, 14, 217—239, 331—358; cf. B., 1927, 86).—V. Using the methods already described, it is shown that tetramethylammonium hydroxide and salts are highly toxic to *A. rumicis*, and that the corresponding tetraethylammonium compounds are markedly less toxic. Certain interesting relationships in regard to toxicity to insects among the substituted anilines and naphthylamines are noted. The heterocyclic rings constituting the molecule of nicotine are much less toxic than is nicotine itself. Hydrogenation of pyridine and pyrrole increases their toxicity. Benzylpyridine is the most toxic pyridine derivative tested.

VI. The toxicity of the fatty acids increases as the series is ascended from acetic to undecic acid, formic acid being exceptional. Beyond this point there is a fall in toxicity, and acids higher than tridecic show only slight toxic action. The sodium and ammonium salts and the methyl esters are, in general, much less toxic than the corresponding acids, the differences being least in the case of the ammonium salts. The fatty acids do not show marked toxicity to certain insect eggs at concentrations below 2%. Possible relationships between certain physical properties (physical state, volatility, dissociation constants, partition coefficients, and surface tension) of the fatty acids and their insecticidal action are discussed.

C. T. GIMINGHAM.

Manufacture of artificial "farmyard manure."

O. LEMMERMANN and E. GERMUND (Z. Pflanz. Düng., 1927, 6B, 481—485).—Experiments are reported on the preparation of artificial farmyard manure by fermentation of straw wetted with dilute solutions of urea and other nitrogen compounds (cf. Hutchinson and Richards, J. Ministry Agric., 1921, 28, 398; B., 1920, 827 A). The product closely resembled well-rotted natural farmyard manure in composition and general appearance.

C. T. GIMINGHAM.

Amount of resins in forest humus and its influence on humification of organic matter. A. NĚMEC (Compt. rend., 1927, 185, 1154—1155; cf. B., 1926, 335).—The formation of humus in the dead surface layer of forest soils and the rate of nitrification vary inversely with the percentage of resins present and inversely with the acidity of the soil. *Oxalis acetosella* and other plants are found where the soil is rich in humus and nitrifying bacteria.

B. W. ANDERSON.

Action of metals [zinc, copper, and iron] on solutions of fungicides. A. WINKELMANN (Z. angew. Chem., 1927, 40, 1393—1394).—The behaviour of solutions of mercurial fungicides, of the strength usually employed, during contact for 30 min. with sheets of iron, copper, and zinc has been investigated. None of the metals has any serious action on "Germisan" or "Segetan-Neu" solutions, iron precipitates practically the whole of the mercury from "Kalimat B" and wheat "Fusariol" solutions, and the greater part from "Urania" and "Uspulun" solutions, zinc precipitates serious amounts of mercury only from the "Fusariol" solutions, and copper is badly corroded by "Urania," precipitates only small quantities of mercury from "Uspulun" and "Fusariol," and is inert in the other cases.

A. R. POWELL.

Jute seeds. SEN.—See XX.

PATENTS.

Increasing the yield of the soil for growing plants and fruits. A. WENDLER (B.P. 251,285, 22.4.26. Ger., 22.4.25).—The yield of crops may be increased by covering the soil with strips of material composed of two layers of weatherproofed paper or the like, having a series of wires fitted between them through which an electric current is passed. The covering is perforated for the growth of the plants in a manner which avoids exposing the wires. C. T. GIMMINGHAM.

Manufacture of an arsenical product [insecticide]. J. F. BLYTH and C. ELLIS (U.S.P. 1,649,562, 15.11.27. Appl., 3.1.25).—Finely-divided arsenious oxide is treated with an excess of quicklime in the presence of just sufficient water to give a substantially dry powder consisting of a mixture of basic calcium arsenite and calcium hydroxide. An insecticide low in water-soluble arsenic is claimed. C. T. GIMMINGHAM.

Copper derivative for combating animal and plant pests. R. LIESKE and W. BONRATH, Assrs. to WINTHROP CHEMICAL CO., INC. (U.S.P. 1,649,536, 15.11.27. Appl., 23.3.25. Ger., 10.4.24).—See F.P. 595,974; B., 1927, 536.

XVII.—SUGARS; STARCHES; GUMS.

Extraction of crystallised sucrose from the carob. G. ONDO and V. DE FONZO (Giorn. Chim. Ind. Appl., 1927, 9, 400).—Industrial extraction of sugar from the carob, which contains 20–26% of sucrose and about 15% of invert sugar, may be effected by the direct action of organic solvents, especially ethyl or methyl alcohol or acetone. The sugar is obtained first as pale yellow crystals with a pleasant carob odour, both colour and odour disappearing after a single crystallisation; the yield of white sugar varies from 16 to 23% of the weight of carobs. Distillation of the mother-liquors leads to the recovery of almost the whole of the solvent, which may be used for subsequent extractions. The residual thick molasses contains dextrose and laevulose, together with 2–3% of sucrose, and may be utilised for the preparation of sweetmeats, alcohol, antiparasitic mixtures for plants, etc. The seeds, amounting to about 10% of the weight of the carob, sell at about 1.2 lire per kg., the perisperm yielding a dyestuff which imparts delicate pink tints to silk and wool, and the albumin a size for dressing and tanning; the residues also are utilisable. Sicily produces 300,000–450,000 quintals of carobs per annum, and the whole of Italy about 800,000 quintals. T. H. POPE.

Adsorption of sucrose by adsorbent carbons. J. VAŠÁTKO (Z. Zuckerind. Czechoslov., 1927, 52, 21–32).—Adsorption equilibrium between active carbons and pure sucrose solutions of concentrations up to 65% was investigated with apparatus which enabled the solutions to be filtered at the equilibrium temperature without evaporation losses. 200 c.c. portions of the sugar solutions, rendered very slightly alkaline, were shaken with 10 g. of carbon previously freed from acidity, dried at 80°, and then exposed to the air, and the change in concentration of the sugar solutions was determined. The results obtained at 20° are accounted for by assuming that both sucrose and water are adsorbed,

each in proportion to the logarithm of its concentration. In the case of Supranorit 3, for example, 1 g. adsorbs 0.302 g. of water from pure water; with increasing concentration of sugar the amount of water adsorbed diminishes logarithmically whilst that of sugar increases, so that 0.575 g. would be adsorbed from a 100% solution. The maximum adsorption of water and sugar together occurs in presence of 40% solutions, and amounts to 0.703 g. per g. of carbon. For the six carbons tested, the relative adsorptive powers for sucrose, at 20°, were: Carbo animalis, 0.954; Supranorit 3, 0.575; Carboraffin, 0.616; Kahlbaum's Spodium, 0.436; Superior Norit, 0.224; Standard Norit, 0.229. The relative water-adsorbing powers were, in the same order, 0.539, 0.302, 0.500, 0.369, 0.225, 0.231. Adsorption of water, or imbibition, is of technical importance, for it increases the volume of the carbon and retards filtration. In general, imbibition will be greater with carbons activated by means of chemicals (impregnation) than with those activated by hot gases. Experiments at high temperatures, up to 80°, indicate that adsorption of sucrose increases slightly with rise of temperature, but adsorption of water decreases considerably. J. H. LANE.

Decomposition of sucrose by adsorbent carbons. J. VAŠÁTKO (Z. Zuckerind. Czechoslov., 1927, 52, 129–137).—Twelve active carbons were treated with dilute alkali, washed until practically neutral, and dried. Quantities up to 18 g. were shaken with 200 c.c. portions of a 20% sucrose solution of p_H 8.0 for 5 hrs. In many cases considerable decomposition of sucrose occurred with formation of invert sugar and acid products. The amount of decomposition varied with the temperature, the character and amount of the carbon used, and the intensity of shaking. At 20° the decomposition was negligible in all cases. At 85° all the solutions containing more than 10 g. of carbon per 200 c.c. became more or less acid, as did some of those containing less. In a number of cases 10–20% of the sucrose was inverted or decomposed. Only slight decomposition was produced by the carbons activated by gaseous treatment, viz., Carbo animalis, the Norit products, and Polycarbon. In marked contrast with this group were Carboraffin, Carboraffin H, blood charcoal, Antichromos, and Kahlbaum's Spodium, most of which are activated by impregnation processes. With few exceptions the members of the first group contain more than 90% C, less than 2% of ash, and less than 0.2% Fe_2O_3 , whilst those of the second group are poorer in carbon and contain more mineral matter and iron. There is no apparent relation between adsorptive power and decomposing action on sucrose. The decomposition is autocatalysed by the acids formed, and is, therefore, greatly reduced by increase of initial alkalinity. J. H. LANE.

Molasses formation and the nature of molasses. H. CLAASSEN (Z. Ver. deut. Zucker-Ind., 1927, 675–678).—The formation of molasses is attributed to combination of part of the sugar with salts present, whereby the ratio of free sugar to water falls below the ordinary saturation ratio. In cane molasses the salts combine by preference with the invert sugar, so that the ratio of sucrose to water corresponds more nearly with that of a pure solution than is the case with beet

molasses. Another factor contributing to molasses formation is the high viscosity, which at low temperatures impedes or prevents crystallisation to the true equilibrium point. Prolonged heating of impure syrups at high concentrations and high temperatures increases the amount of sugar-salt compounds and so diminishes the recoverable sugar. Hence in sugar-boiling it is best to work quickly and at as low a temperature as possible, and to limit the re-boiling of syrups; three rapid boilings are preferable to two slow ones. Another effect of prolonged boiling, not detectable by analysis or viscosity measurements, is to increase the temperature difference necessary between the heating steam and the evolved steam; probably the sugar-salt compounds impede the formation of bubbles and so raise the temperature required for ebullition under a given pressure.

J. H. LANE.

Trimethylamine in the vapours from carbonatation [of beet juices]. J. DĚDEK (Z. Zuckerind. Czechoslov., 1927, 52, 33—35).—An account of an attempt to identify and determine the volatile bases which accompany ammonia in the vapours from beet sugar factory juices. These bases represent less than 1% of the total nitrogen of the volatilised matters. Only trimethylamine was isolated in the pure state. No traces of mono- or di-methylamine were found, but more complex bases of the pyridine group were present although not identified.

J. H. LANE.

Determination of the crystal content of raw sugars. O. SPENGLER and C. BRENDL (Z. Ver. deut. Zucker-Ind., 1927, 679—689).—The method recently described (B., 1927, 730) gives the amount of technically recoverable crystals in raw sugars, but for accurate determinations of the total crystal content a modified method is now described in which the raw sugar is mixed with syrup saturated at the temperature of the test instead of 3° below, and a felt or flannel pad is placed in the centrifugal apparatus to retain fine grain. The new method is thus identical in principle with that of Herzfeld and Zimmermann (B., 1912, 244), but the apparatus is smaller and somewhat different in design.

J. H. LANE.

Determination of ash in raw [beet] sugars by incineration and by the electrometric method. F. HERLES (Z. Zuckerind. Czechoslov., 1927, 52, 145—148).—The electrometric determination of ash in beet sugars by means of Sandera's apparatus (B., 1927, 312) gave results which in 108 out of 127 cases enumerated agreed to within 0.03% with the values found by the usual method of incineration. In the remaining 19 cases the differences were due to the presence of matter insoluble in water and therefore without melassigenic action. With few exceptions the sugars tested contained less than 1.3% of ash.

J. H. LANE.

Determination of ash in raw [beet] sugars. C. MRASEK (Z. Zuckerind. Czechoslov., 1927, 52, 149).—Results for 21 first-product beet sugars confirm the close agreement between the results of the electrometric and the incineration methods, reported by Herles (preceding abstract) and others. The average difference for the 21 samples was 0.007%.

J. H. LANE.

Determination of melassigenic nitrogen in beet-

root. M. S. FILOSOFOV (Ukraine Chem. J., 1926, 2, 127—135).—The total soluble nitrogen in beetroot was first determined and then that present as amides and ammoniacal salts, the difference giving the amount of melassigenic nitrogen—*i.e.*, that present as betaine and primary amines. The values so obtained agreed within 0.04% with those found by direct titration with formalin. In the case of treacle it is necessary to acidify the solution before titration until an acid reaction towards litmus is given.

A. RATCLIFFE.

Control of spectrophotometric measurements [of sugar juices]. H. LUNDÉN (Z. Ver. deut. Zucker-Ind., 1927, 709—713).—A reply to criticisms of a recent paper (B., 1927, 395) by Spengler and Landt (*ibid.*, 664). The Lambert-Beer law of absorption holds only for perfectly clear solutions, and cloudiness is much more difficult to eliminate from cane than from beet sugar products. In refinery working the yield of sugar obtainable from a massecuite can be calculated if two suitable analytical values are known for the massecuite itself, and for the sugar and syrup obtained by centrifuging. Using as analytical values the dry-substance content and the spectrophotometric absorption, the author calculated yields from affination and from massecuite working which agreed to within 3% with the actual yields of sugar found by weighing.

J. H. LANE.

Clarification of starch conversion liquors in manufacture of corn sugar and corn syrup. M. S. BADOLLET and H. S. PAINE (Ind. Eng. Chem., 1927, 19, 1245—1246).—A method for the flocculation of the colloidal material in acid starch conversion liquors is described. Since the colloid particles have a positive electric charge, coagulation is produced by the addition of negatively charged colloids such as bentonite, colloidal aluminates, colloidal alkaline compounds of iron, silicates of aluminium, etc., together with sodium carbonate to reduce the acidity. The results obtained by clarification with sodium carbonate alone and with bentonite and sodium carbonate are compared. The latter method, after correcting for the weight of bentonite present, gave an increase in weight of flocculated precipitate over that obtained by sodium carbonate, varying from 3 to 258%. The colloidal material flocculates and settles more rapidly, and the liquor is better prepared for treatment with activated carbon. Of the nine converter liquors examined, the amount of bentonite per 1000 gals. of liquor, *i.e.*, the amount required to reach the isoelectric point, varied from 0.8 to 23.3 lb. (cf. B., 1926, 507).

E. H. SHARPLES.

Decomposition of starch by the amylase of *Aspergillus oryzae* and of malt with special reference to the limit of decomposition. S. NISHIMURA (Woch. Brau., 1927, 44, 533—535).—When starch is acted on by commercial taka-diastase (from *Aspergillus oryzae*) or by an extract from malt, over 95% may be hydrolysed provided a sufficiently large amount of enzyme is employed. Under these conditions the normal limit of decomposition (70—80%) is exceeded independently of the action of "complement." W. O. KERMACK.

Purity quotient of [sugar] beet juice. O. SPENGLER and C. BRENDL (Z. Ver. deut. Zucker-Ind., 1927, 747—752).

Adhesion of gums. MCBAIN and LEE.—See I.

Refractometer for sugar. LÖWE.—See I.

PATENTS.

Decolorisation of sugar crystals. RAFFINERIE TIRLEMONTAISE SOC. ANON. (B.P. 278,302, 2.11.26. Ger., 30.9.26).—Sugar crystals which have undergone a preliminary washing to free them from the adhering syrup, but which are still coloured, are mashed with a colourless saturated or nearly saturated sugar solution for a period of time depending on the depth of colour. The crystals are then separated from the solution by centrifugal action or other means, and the separated solution which has absorbed the colouring matter is decolorised by filtration through charcoal and used again.

F. R. ENNOS.

Curing of massecuite. RAFFINERIE TIRLEMONTAISE SOC. ANON. (B.P. 274,799, 2.11.26. Ger., 24.7.26).—An increased yield of sugar crystals is obtained from massecuite by subjecting it to prolonged cooling and removing a part of its water content by evaporation, either during the cooling process or on its completion. The resulting pasty or plastic mass is then separated into sugar crystals and syrup by centrifuges operating at a considerably greater centrifugal force (1500—1600 times the weight of the mass) than those previously used for the purpose.

F. R. ENNOS.

Removal of false grain from sugar syrups or molasses. RAFFINERIE TIRLEMONTAISE SOC. ANON. (B.P. 274,800, 2.11.26. Ger., 24.7.26).—The sugar syrup or molasses is (a) subjected to an extremely high centrifugal force (1800—8000 times the weight of the mass treated, depending on the viscosity of the syrup and the size of the false grain) and the false grain collected on the unperforated casing of the centrifuge; or (b) mixed, before centrifuging, with air or other gas in a fine state of division and the false grain separated with the froth formed on the upper surface of the mass after centrifuging.

F. R. ENNOS.

Porous materials from sugar-factory residues (B.P. 280,116).—See IX.

Polyamyloses (G.P. 442,963).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Transformation of the α -bitter acid of hops (humulone) on boiling in aqueous solutions of different p_H values, and the nature of the products formed. W. WINDISCH, P. KOLBACH, and R. SCHLEICHER (Woch. Brau., 1927, 44, 453—459, 473—478, 485—490, 497—502).—Humulone, the α -bitter acid of hops, when boiled with malt wort or aqueous solutions not far removed from neutrality, is converted into a mixture of soft resins, soluble in light petroleum, and hard resins insoluble in this solvent. The hard resins are oxidation products and are not formed if air is rigorously excluded. The soft resins are intermediate products in the transformation of humulone into humulinic acid, a transformation which proceeds to completion on boiling with moderately strong alkali. In slightly acid liquids, such as wort, only about a third of the humulone is converted into soft resins by 2 hrs.' boiling, and scarcely any humulinic acid is formed. The soft resins are probably

a mixture of two compounds, one isomeric with humulone and the other of lower mol. wt. J. H. LANE.

Influence of temperature of storage of yeast under water on fermentation, reproduction, and acid-formation in wort. F. STOCKHAUSEN and F. WINDISCH (Woch. Brau., 1927, 44, 478—481).—If bottom-fermentation brewery yeast from the vats, after being sifted and washed, is kept under cold water until required for later fermentations, the temperature of the water has an influence on the vigour of the yeast. Comparison of the effects of storage for three days under water at 1°, 4°, 7°, and 18° showed that the lower the temperature the more rapid were fermentation and reproduction during the first few days of the subsequent fermentations, but by the end of the full fermentation period (nine days) the degree of attenuation, the weight of the yeast crop, and the wort acidity were practically the same in all cases. J. H. LANE.

Tartaric acid index in red and white pressed wines. FOXZES-DIACON (Ann. Falsif., 1927, 20, 467—471).—A red, pressed wine may have a very similar content of alcohol and acid to the corresponding unpressed wine. The two classes of wine may be distinguished by means of the tartaric acid index, which is greater than 1 for the unpressed wine and less than 1 for the pressed wine; in addition, the pressed wine has a larger extract and ash, and a smaller total of fixed acid and alcohol. The differences in composition between the pressed and unpressed white wines are somewhat greater, and the former might easily be mistaken for a diluted wine were it not for the fact that it has the characteristics of an abnormal wine (cf. B., 1926, 104; 1927, 24), viz., potash calculated as cream of tartar greater than 4, tartaric acid index less than 1, volatile acidity less than 1. The relatively high alcohol and potash content, as well as the low percentage of tartaric acid, in pressed wines is probably accounted for by the fact that there is a larger amount of sugar and a smaller amount of tartaric acid in the outer than in the inner portion of the grape before complete ripening, whilst in pressed wine a large proportion of potash is derived from the stalks. F. R. ENNOS.

***l*-Glutimic acid as nitrogenous nutriment for yeast.** A. DOLINEK (Z. Zuckerind. Czechoslov., 1927, 52, 35—43).—Among the nitrogenous constituents of beet molasses *l*-glutimic acid, the lactam of α -amino-glutaric acid, is second only to betaine in quantitative importance. Raw sugar molasses contain more than 2% and sometimes 5—8%. Molasses from refineries working beet sugar usually contain rather more than raw sugar molasses of the same campaign, probably because prolonged heating of molasses converts part of the glutamic acid present into *l*-glutimic acid. The nitrogen of glutamic acid is more readily assimilated by yeast than that of ammonium sulphate, but glutimic acid is much inferior to either in this respect. This may account in part for the fact that the nitrogen of beet molasses from refineries is sometimes less assimilable than that of raw sugar molasses. J. H. LANE.

Melibiose. I. R. WEIDENHAGEN (Z. Ver. deut. Zucker-Ind., 1927, 696—708).—Melibiose is present in bottom- but not in top-fermentation yeasts. From

the former it can be quantitatively extracted after autolysis with toluene if the liquid is maintained neutral by addition of ammonia as required. Extracts so obtained are much more active than those from dried yeast. Their hydrolytic action on melibiose solutions does not proceed as a unimolecular reaction, the velocity declining too rapidly. The optimum reaction is at p_H 5.0. Yeast extracts obtained as described have the power of decomposing α -methylgalactoside (cf. Hérisséy and Aubry, B., 1914, 272). J. H. LANE.

Influence of certain factors on formation of empyreumatic oil by fermentation of molasses. S. JANKOVIĆ (Arhiv Hemiju, 1927, 1, 218—226).—The yields of empyreumatic oil from the alcoholic fermentation of molasses increase with temperature and concentration of molasses, and fall with increasing acidity of the culture medium. With dilute solutions the addition of large quantities of yeast causes increased production of oil, whilst with concentrated solutions the yield of oil is greater when smaller quantities of yeast are used. R. TRUSZKOWSKI.

Dehydration of aqueous-alcoholic liquids. P. BRUN (Compt. rend., 1927, 185, 1132—1134).—The method of Gay and Massol (cf. B., 1924, 802) has been applied in the light of the author's studies of mixtures of ethyl and isoamyl alcohols and water, the principal constituents of fusel oils (A., 1926, 895). Mixtures containing less than 92% of amyl alcohol should be treated with potassium carbonate after the mixture of the alcohols. In other cases the separated ethyl alcohol should be partly dehydrated with potassium carbonate and, after removal of the saline layer, added to the pure isoamyl alcohol (purified by simple rectification). J. GRANT.

Power alcohol from waste vegetable materials. ANON.—See II. **Decomposition of starch by amylase.** NISHIMURA.—See XVII.

PATENTS.

Treatment of [alcoholic] liquids containing tasty and aromatic substances for the purpose of improving their taste and aroma. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (B.P. 259,598, 7.10.26. Ger., 12.10.25).—Alcoholic liquids may be "aged" by withdrawing a portion of the liquid, subjecting it while under pressure to the action of very rapidly moving surfaces, e.g., the blades of a rotor rotating at high speed, with substantially complete exclusion of air, returning the treated portion to the main body of the liquid, and continuing the process until the desired effect has been obtained. According to the nature of the liquid it may be cooled, heated, or alternately cooled and heated during the treatment, and the internal friction of the liquid and the heating effect produced by the action of the rapidly moving surfaces may be increased by the presence of substances dissolved or finely distributed in the liquid.

F. R. ENNOS.

Fermentation at constant temperature. C. O. BERTIN (F.P. 615,532, 3.5.26).—The number of yeast cells in a given quantity of liquid is artificially reduced by mechanical means, or by the addition of a definite quantity of fermented liquid or of must from which the

yeast has been removed completely or partially after the beginning of the fermentation. In this way the heat losses due to radiation, convection, etc. are balanced by the heat given out by the fermentation process, so that the temperature remains constant. F. R. ENNOS.

Dehydration of alcohol by distillation. U.S. INDUSTRIAL ALCOHOL CO., Assees. of D. B. KEYES (B.P. 268,728, 29.11.26. U.S., 30.3.26).—A continuous process for the removal of water from alcohol by means of benzene, ethyl acetate, carbon tetrachloride, hexane, etc. is described, the essential novelty being that after separation of the two layers forming the condensate, the aqueous-alcoholic layer poor in benzene (etc.) is diluted with sufficient water to cause a further separation of benzene (etc.) before passing to the rectifier. Alternative arrangements of plant are figured suitable for use with (a) benzene, ethyl acetate, hexane, (b) carbon tetrachloride. About equal amounts of benzene and 95% alcohol are introduced into a plate-column heated at 79° at the bottom and kept at about 65° at the top. Absolute alcohol is run off through a trapped pipe at the bottom; the vapours of constant-boiling ternary mixture pass from the top of the column through a dephlegmator to a condenser, and the condensate is collected in a separating chamber. The benzene layer overflows into a rectifying column, where vapours of ternary mixture are evolved and returned by way of a dephlegmator and condenser to the separating chamber; pure benzene is drawn off from the bottom of the rectifying column and pumped back to the alcohol-dehydrating column. The aqueous-alcoholic layer (containing some benzene) is diluted with water in a second separating chamber, the upper layer passing to the benzene-rectifying column, and the lower layer to an alcohol-recovery column. Here the remaining benzene passes off in vapour of ternary mixture, leaving behind aqueous alcohol of about 30% strength, which, after rectification to 95% in a final column (water being run off), returns to the original dehydrating column. C. HOLLINS.

Dehydration and rectification of alcohol and other volatile products. G. BAUGÉ and T. ÉPAILLY (F.P. 615,732, 6.5.26).—The alcohol is intimately mixed with the dehydrating material, consisting of powdered copper sulphate and an inert non-porous material, e.g., sand, silica, etc., both of which have been freed from water (including that of crystallisation). The process is repeated as many times as is necessary with fresh portions of dehydrating material, and the alcohol is subsequently distilled; the dehydrating material is freed from water for further use. F. R. ENNOS.

Butyl [alcohol]-acetone fermentation. COMMERCIAL SOLVENTS CORP., Assees. of D. A. LEGG (B.P. 278,307, 28.3.27. U.S., 1.10.26).—A normal yield of solvents is obtained from carbohydrate fermentation, regardless of the presence of an ultravirus or bacteriophage in the mash, by rendering the butyl-acetone bacilli resistant to epidemics of sluggishness. The immunisation is accomplished by repeatedly subcultivating the bacilli in a sluggishly fermenting mash or in a Berkefeld filtrate therefrom, the cultures being heated for 3 min. at 100° before each transfer in order to eliminate attenuated vegetative cells (cf. U.S.P. 1,582,408; B., 1926, 563).

C. RANKEN.

Isolation of polyamyloses. I. G. FARBERIND. A.-G., Assees. of F. LANGE (G.P. 442,963, 25.1.25).—The solution obtained by fermentation of starch or starchy materials with bacteria is treated with a halogen derivative of the ethylene series. The additive compounds of polyamyloses separate in easily filterable form.

C. HOLLINS.

XIX.—FOODS.

Influence of the age of a milk on the result of pasteurisation. A. WOLFF (Milch. Zentr., 1927, 11, 233—238).—Cultures on gelatin and agar-agar were made of samples of fresh milk and of the same milks after sterilising by heating for $\frac{1}{2}$ hr. at 62–63°, followed by rapid cooling, and a comparison was made of the total number and of the relative proportions of the different kinds of bacteria in the fresh and sterilised milks. Similar tests were made on the milk after keeping for periods varying from 2 to 8 hrs. at temperatures from 8° to 30°. The results show that the number of bacteria surviving pasteurisation, as well as the percentage of these calculated on the total number present in the unpasteurised milk, are greater for the milks which have been kept for a time than for those which were sterilised at once. In practice, it is therefore preferable to sterilise a milk as early as possible and then to keep it cool, rather than to keep it in cold storage and sterilise later.

F. R. ENNOS.

Composition of the fat of mare's milk. O. LANA (Ann. Falsif., 1927, 20, 462–464).—The fat extracted from a sample of mare's milk had m.p. 8–9°, f.p. 5–6°, saponif. value 227.8, Reichert–Meissl value 7.0, Wauters–Polenske value 6.1. The composition of the fat was caprylic acid 2.29%, capric acid 2.19%, oleic acid 67.80%, the remaining 27.72% consisting of lauric, myristic, and palmitic acids, and glycerol. The milk fat, unlike that of ruminants, contained no butyric or caproic acids.

F. R. ENNOS.

Determination of lactic acid in milk. J. STRAUß (Rec. trav. chim., 1927, 46, 866–884).—Lactic acid is determined in milk, which has been freed from casein and lactose, by distilling with 1% of sulphuric acid and 0.02N-potassium permanganate, collecting the distillate in standardised potassium hydrogen sulphite, and determining the excess with iodine solution. Other constituents which affect the freezing point of milk (chlorides and lactose) are determined and their total contribution to the observed depression is calculated. The difference between this figure and the observed depression is due to substances of unknown nature, and is large in the milk from cows suffering from udder disease, but is also present in normal milk.

G. A. C. GOUGH.

Use of quinhydrone electrode for following changes of p_H in Swiss cheese. P. D. WATSON (Ind. Eng. Chem., 1927, 19, 1272–1274).—For control of the cheese-making process, a combination of the capillary quinhydrone electrode of Cullen and Biilmann (A., 1925, i, 1201) and the cheese quinhydrone electrode of Knudsen (B., 1926, 105) is employed, the former for the 3- or 4-hr. period after dipping (i.e., on the whey) and the latter on samples of the cheese itself during the remainder of the run. Whereas the hydrogen

electrode can only be used for 3 hrs. owing to the difficulty of obtaining sufficient whey, the method described allows the changes in p_H value to be followed during the whole process. Identical values were obtained with the three types of electrodes on two unrelated cheeses during the first 3 hrs. The method is fully described and the effect of varying the cooking temperature, type of starter, and variation in percentage of starter on the p_H of the cheese during manufacture has been examined.

E. H. SHARPLES.

Effect of temperature on the basic viscosity of ice-cream mixes. A. LEIGHTON and O. E. WILLIAMS (J. Physical Chem., 1927, 31, 1663–1668; cf. B., 1927, 375).—The basic viscosity of different water concentrations of two mixes of essentially the same composition has been found over the temperature range 8° to –1°. The viscosity varies linearly, inversely as the temperature. The viscosity of the unfrozen portion of the mix increases during the freezing process, which brings about an increase in concentration of the milk solids and sugar in the liquid phase.

L. S. THEOBALD.

Composition, constitution, and food value of residues from the grinding of wheat. J. ALQUIÉR (Ann. Falsif., 1927, 20, 445–462).—The bran residues in modern milling practice fall into three or four main classes, the exact number, nomenclature, and fineness of division of each residue of which vary with the source of supply and methods of grinding employed. Microscopical examination reveals no predominant structure in any one type, each being a mixture of the pericarp, germ, and small amounts of adhering flour. Chemical analysis shows that the proportion of starch diminishes with increase in the pentosan and the cellulose content, and the ratio of starch to pentosan or of starch to cellulose varies regularly from one type to another, having values which within fairly wide limits are characteristic of each. Biological analyses based on the period of survival of rats fed solely on these residues serve to differentiate them into two distinct classes. The suitability of the residues as constituents of stock foods is due not only to the adhering flour, but also to their high content of amino-acids and vitamins. 100 pts. of corn yield 74.5 pts. of flour for bread-making, 6 pts. of flour adhering to the residues, 16 pts. of residues (without flour), 1.5 pts. of germ, and a 2% milling loss.

F. R. ENNOS.

Application of p_H theory to the conservation of grain and cereal by-products. H. LEGENDRE (Compt. rend., 1927, 185, 1156–1158).—The addition of small quantities of harmless alkaline materials to grain in storage or transport, by altering the p_H value, successfully inhibits the formation of sugars by the action of diastases on starch from the split grains. The absence of sugars prevents the multiplying of bacteria and thus obviates the usual rise in temperature with its attendant evils of germination and fermentation.

B. W. ANDERSON.

Behaviour of certain varieties of pears towards cold storage. E. BOTTINI (Annali Chim. Appl., 1927, 17, 457–486).—Pears of certain varieties cultivated in Italy may be stored under suitable conditions at 1–2°. Different kinds require different degrees of atmospheric

moisture and different rates of ventilation. Pears thus treated contain no appreciable amount of sucrose when mature, but otherwise scarcely differ from those ripened naturally, although their weight is diminished by about 10%. "Internal breakdown" sometimes occurs, but may be minimised by gathering the fruit in the proper condition, and "scald" may be avoided by promoting the elimination of the volatile esters by suitable ventilation. T. H. POPE.

Barbary figs. A. AZADIAN (Ann. Falsif., 1927, 20, 464—467).—Analyses of a number of samples of the fruit from different sources show that it is composed on the average of 40% of rind, and 60% of fruit containing 82% of pulp and 18% of seeds. The pulp contains 8.66—12.36% of solids, 0.28—0.73% of ash, 0.05—0.81% of acidity, 0.20—0.70% of proteins, and 8.06—10.22% of sugars. On extraction with ether and light petroleum, the seeds yield 5.8% of an oil which has d_{4}^{25} 0.9253—0.9493, saponif. value 193—205, acid value 3—6, and iodine value 102.8—117.4. F. R. ENNOS.

Nutritive value of sugar beet tops. WOODMAN and BEE.—See XVI.

PATENTS.

Preparation of flavouring, nourishing, and similar substances from proteins, carbohydrates, or other animal or vegetable matter. M. FUJII (B.P. 279,985, 11.9.26).—Animal or vegetable matters such as proteins or carbohydrates are decomposed to peptones or sugars respectively by treatment with alkali or acid, and the resulting product is neutralised by glutamic acid or its salts produced by treating animal or vegetable proteins in an autoclave with a catalyst (powdered iron or iron oxide) and mineral acid and heating to the usual temperature and at a pressure not exceeding 4 atm. The disodium or other metallic salts are used for neutralisation after the acid treatment, and glutamic acid or its hydrochloride after the alkaline treatment, the quantity of each which is used being sufficient to produce the monosodium salt of glutamic acid. The neutralised product may be concentrated or dried with or without the addition of one or more medicinal substances, foods, or flavours. F. R. ENNOS.

Method and apparatus for curing [maturing] flour. E. C. R. MARKS. FROM INDUSTRIAL APPLIANCE Co. (B.P. 279,958, 10.8.26).—A mixture of chlorine with a small quantity of nitrosyl chloride gas is diluted with air or other inert gas and forced through dilute aqueous ammonia contained in a reaction tower, the chlorine and nitrosyl chloride being in excess of the amount which will combine with the ammonia. The tower is filled with glass balls or similar objects to ensure intimate mixing of the gases resulting from the reactions, which are then brought into contact with the flour in a dispersed state in a treating chamber. F. R. ENNOS.

Preservation of eggs. A. ANDRIEU (F.P. 619,375, 28.11.25).—The eggs are cooled to -1° and placed in a closed, air-tight container into which, after removal of the air, a mixture of carbon dioxide and nitrogen is introduced. After allowing sufficient time for the eggs to absorb the inert gases, they are removed and stored at $0-1^{\circ}$. F. R. ENNOS.

Preservation of feeding stuffs. J. TAUFER, V. PAVLAK, and M. BAYER (F.P. 613,213, 22.3.26. Conv., 6.4.25).—Green fodder, beet parings, etc. are placed in an air-tight container, *e.g.*, of concrete, which has a lid with a rim dipping into a liquid seal consisting of water, oil, or a mixture of water and glycerin. While in the container the feeding stuffs undergo lactic acid fermentation with evolution of carbon dioxide which escapes through the liquid seal, and the final product is preserved and protected from further decomposition by the absence of air. F. R. ENNOS.

Preservation of green fodder containing sap. B. DIRKS (G.P. 443,884, 12.1.23).—Green fodder may be economically, simply, and safely preserved by treating layers of the material, pressed for storage purposes, with a dilute solution of formic acid. F. R. ENNOS.

Manufacture and preservation of green fodder pulp or other green plant pulp and of dry products made therefrom. K. EREKY (B.P. 270,629, 28.9.26. Hung., 7.5.26).—Green plants are comminuted by a cutting apparatus, so as to disrupt the individual cells and set free the fluid plasm therefrom. The fluid pulp produced, which contains fragments of cell walls but no intact cells, may be separated into cell juice and cell-wall residues by sieving, centrifuging, etc., and brought into condition for storage by cooling or treating with disinfectants. F. R. ENNOS.

Production of [stabilised] vitamins. C. FUNK and H. E. DUBIN, Assrs. to H. A. METZ LABORATORIES, INC. (U.S.P. 1,649,520, 15.11.27. Appl., 13.5.22).—The vitamin content of materials may be stabilised by mild hydrogenation of the material at a temperature insufficient to destroy the vitamin. The stabilised vitamin may be separated. *E.g.*, a vitamin concentrate may be obtained from cod-liver oil by emulsification in water, hydrogenation in the presence of colloidal platinum, separation of the hydrogenated product, and crystallisation from chloroform. The product is extracted with alcohol and ether, and the residue purified by the digitonin precipitation. B. FULLMAN.

Production of pure white pectin. R. PAUL and R. H. GRANDSEIGNE (F.P. 614,882, 22.4.26).—Pectin is precipitated from solution by the addition of acetone and the product is washed repeatedly with acetone. L. A. COLES.

Production of leavened dough products. T. B. WAGNER and C. A. GLABAU (U.S.P. 1,649,144, 15.11.27. Appl., 21.6.24. Renewed 5.4.27).—See B.P. 235,874; B., 1927, 154.

Apparatus for treating [pasteurising] milk etc. A. G. ENOCK & Co., LTD., and E. C. ENOCK (B.P. 280,328, 21.9.26.)

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Production of saponins from horse-chestnuts. R. VADAS (Chem.-Ztg., 1927, 51, 895).—Horse-chestnuts are defatted with light petroleum, extracted with 70% alcohol, and the extract is evaporated under reduced pressure. The residue is dissolved in alcohol and the raw saponin, containing 5—6% of ash, is precipitated by the addition of ether. The product

is heated with 6 pts. of acetic anhydride in presence of zinc chloride; the acetylated saponin is collected on a filter and mixed with freshly precipitated lead hydroxide, whereby a snow-white, ash-free saponin is produced which is removed by filtration and dried in a vacuum (cf. B., 1924, 993; 1925, 469).

E. H. SHARPLES.

Determination of morphine when present in small quantities. H. BRINDLE (Pharm. J., 1927, 119, 608—609).—The method proposed by Nicholls (B., 1922, 159 A) and recommended by Caines (B., 1927, 570) is found to be accurate both for small and large quantities. Free alkali hydroxide must be neutralised before the addition of ammonia. For quantities less than 0.02 g. the colorimetric method with potassium iodate is employed; in this case it is essential that the standard colours and the morphine solution should be prepared under exactly the same specified conditions.

S. I. LEVY.

Rapid determination of urotropine. E. OLIVERI-MANDALA and G. RICCARDI (Annali Chim. Appl., 1927, 17, 487—490).—About 0.5 g. of the urotropine is boiled with 40 c.c. of *N*-sulphuric acid until all odour of formaldehyde disappears (about 30 min.). The cold liquid is diluted with water and the unused acid determined by titration with *N*-sodium hydroxide in presence of Methyl Orange, the amount of urotropine in the sample being calculated in accordance with the equation: $(\text{CH}_2)_6\text{N}_4 + 6\text{H}_2\text{O} + 2\text{H}_2\text{SO}_4 = 6\text{CH}_2\text{O} + 2(\text{NH}_4)_2\text{SO}_4$. The result may be checked by adding excess of formaldehyde to the neutralised solution, so as to effect the reverse reaction to the above, and determining by titration the sulphuric acid liberated. Disagreement between the two results indicates the presence in the original urotropine of ammonium salts, the amount of which is determined by the excess of alkali required in the second titration.

T. H. POPE.

Reactions of anæsthetic ethers with potassium hydroxide and with mercury, and the test for foreign odours. E. MALLINCKRODT, JUN. (J. Amer. Chem. Soc., 1927, 49, 2655—2666).—The method of the U.S.P. X. test for aldehyde in ethyl ether is normally not sensitive to less than 0.05% of aldehyde; 0.01% of aldehyde may be detected by pouring 30 c.c. of the ether on to 5 g. of freshly-broken potassium hydroxide stick, adding 1 drop of water, and keeping the mixture in the dark for up to 12 hrs., when a brown colour is produced on the surface of the potassium hydroxide and a paler tint in the solution. If alcohol is present a brown coloration, due to this impurity, may develop on the potassium hydroxide in 12 hrs., so that the test for aldehyde should be examined after 6 hrs. Disturbance of the aldehyde test due to alcohol is reduced by the addition of water. Thus anhydrous ether containing 1.42% of alcohol becomes turbid and yellowish in contact with dry potassium hydroxide in 6 hrs., but remains clear for 24 hrs. if 3 drops of water are added in the test. The test is unreliable in the presence of peroxides unless the amount of the latter is very small. Shaking the ether with metallic mercury decomposes the peroxides with liberation of aldehyde, but probably not quantitatively. The presence of considerable amounts of per-

oxides and alcohol is indicated by the appearance of a turbidity which remains constant in intensity for some hours, but this turbidity is not easily distinguishable from that due to alcohol alone. If the ether contains several per cent. of alcohol together with about 0.1% of acetaldehyde, there is formed in the above test a precipitate resembling ferric hydroxide. Acetaldehyde and furfuraldehyde behave similarly to acetaldehyde. Traces of paraldehyde produce no coloration unless previously decomposed by warming the sample with a drop of sulphuric acid. By evaporating 30 c.c. of ether slowly in a beaker covered with a watch-glass to 1—2 c.c., and pouring the residue slowly on to filter paper, a final residue is obtained the odour of which affords a delicate test for impurities. The presence of alcohol is thus made manifest, whilst water retards the evaporation on the paper. Peroxides form an oily spot at the centre of the paper or at the margin of the evaporating liquid, and then produce a characteristic irritation when placed close to the nostrils.

F. G. WILLSON.

Jute seeds—*Corchorus Capsularis*. I. N. K. SEN (J. Indian Chem. Soc., 1927, 4, 205—208).—A preliminary investigation of jute seeds, used medicinally in India in cases of fever, is made. Extraction of the seeds with light petroleum yields a yellow oil (14.7%; d_{20}^{25} 0.921; $[\alpha]_D^{25} \pm 0$; iodine value, 109.2; acid value, 24.07) which dries in air to a rubber-like mass. The oil is thought to be a mixture of glycerides of unsaturated fatty acids. The extracted seeds lose their bitter taste when re-extracted with spirit—a process which yields a substance which separates from acetone and glacial acetic acid in white, hygroscopic crystals (shrinking at 98° and decomposing at 105°, $[\alpha]_D^{25} + 103.6^\circ$; found, C, 31.16%; H, 9.65%). This substance has a bitter taste and corresponds in chemical properties with a glucoside. From the mother-liquors another colourless substance with a very bitter taste is obtained in small amount.

G. A. C. GOUGH.

Essential oil of *Eucalyptus Bakeri* (Maiden). A. R. PENFOLD (J. Proc. Roy. Soc. New South Wales, 1927, 61, 179—189).—Examination of the oils, obtained in 1% yield on freshly cut leaves, and 2% on air-dried, from four samples of *E. Bakeri*, shows the presence of the following substances: cineole (70—77%), together with small amounts of cymene, *d*- α -pinene, phloracetophenone dimethyl ether, m.p. 82—83° (from one sample only), *p*-isopropylphenol, cuminal, cryptal, phellandral, cuminal, phellandrol, and formic, isobutyric, and isovaleric acids (free and combined).

H. BURTON.

Biological assay of aconite. M. G. JAUREGUI (J. Amer. Pharm. Assoc., 1927, 16, 1045—1052).

Refractometer for sugar. LÖWE.—See I.

Calcium citrate. MELIS.—See VII.

Contact insecticides. TATTERSFIELD and GIMINGHAM.—See XVI.

PATENTS.

Preparation of ethyl chloride from ethyl alcohol. I. G. FARBENIND. A.-G., Assees. of K. DACHLAUER and E. EGGERT (G.P. 441,747, 15.2.24).—Ethyl alcohol vapour and hydrogen chloride are passed over a phos-

phoric acid-active carbon catalyst at 280–300°. The yield (75% with ordinary active carbon) is 97%.

C. HOLLINS.

Preparation of hydroxyalkylamides of o-hydroxybenzoic acids. I. G. FARBERIND. A.-G., Asses. of W. KROPP (G.P. 442,038, 13.12.24).—Salicylic and cresotic hydroxyalkylamides, obtained by the usual methods, have powerful antirheumatic and antineuralgic action. *Salicylic β-hydroxyethylamide*, m.p. 119°, prepared from β-aminoethyl alcohol and methyl salicylate or o-acetoxycarbonyl chloride (with subsequent hydrolysis), *salicylic βγ-dihydroxy-n-propylamide*, and *m-cresotic β-hydroxyethylamide*, m.p. 88°, are described. C. HOLLINS.

Preparation of water-soluble, easily hydrolysed benzyl derivatives. AUGUSTE-VICTORIA-APOTHEKE REHWALD & WEISS, and H. OHLE (G.P. 441,463, 10.5.25; cf. Hintzelmann, Joachimoglu, and Ohle, A., 1926, 288).—Benzyl esters of aliphatic and aromatic hydroxy-acids are esterified with chlorosulphonic acid, e.g., in pyridine at –10°, to give sulphato-compounds which form water-soluble sodium salts. *Benzyl sodiosulphatoglycolate*, $\text{CH}_2\text{Ph}\cdot\text{O}\cdot\text{CO}\cdot\text{CH}_2\cdot\text{O}\cdot\text{SO}_3\text{Na}$, sinters at 90°, fully melted at 170° (potassium, calcium, and barium salts also described), and benzyl sodiosulphatomandelate ("betilon," loc. cit.) are obtained from benzyl glycolate and mandelate, respectively. C. HOLLINS.

Manufacture of 1:3-di[hydr]oxy-4-carboxy-6-phenylethylbenzene [2:4-dihydroxy-αβ-diphenylethane-5-carboxylic acid]. F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 272,232, 7.6.27. Ger., 4.6.26).—2:4-Dihydroxy-αβ-diphenylethane, obtained by reduction of the condensation product of resorcinol with phenylacetonitrile, is carboxylated by heating with aqueous sodium hydrogen carbonate at 140° under pressure, or by the action of carbon dioxide on the dry sodio-derivative (prepared by means of sodium ethoxide) at 100–120° under pressure. The 2:4-hydroxy-αβ-diphenylethane-5-carboxylic acid, m.p. 190° (decomp.), so produced has strong germicidal properties even in neutral solution and passes unabsorbed through the human body. C. HOLLINS.

Preparation of means for fighting bacterial diseases. C. RÄTH (B.P. 262,080, 10.11.26. Ger., 24.11.25).—Virulent bacterial cultures are inoculated with non-alkaloid derivatives of pyridine or quinoline (e.g., 5-iodo-2-hydroxypyridine), or such derivatives are added to a nutrient broth on which the culture is then grown, or a culture is washed with salt solution, centrifuged, and the residue treated with the pyridine or quinoline derivative in salt solution. Vaccines are so obtained which are both prophylactic and curative; they may also be used in the processes of B.P. 247,965 (B., 1927, 619). C. HOLLINS.

Manufacture of n-propyl ester of 2-phenylquinoline-4-carboxylic acid. A. J. STEPHENS. From J. A. VON WÜLFING (B.P. 279,745, 17.6.27).—*n-Propyl 2-phenylquinoline-4-carboxylate*, m.p. 63°, obtained by the usual esterification methods, is non-irritant and tasteless, and dissolves uric acid in the human body more readily than methyl, ethyl, or allyl esters. C. HOLLINS.

Production of 6-chlorothymol. F. RASCHIG (B.P.

270,283, 21.4.27. Ger., 29.4.26. Cf. G.P. 433,293; B., 1927, 237).—6-Chloro-*m*-cresol is condensed with isopropyl alcohol in presence of sulphuric acid at 80–90° giving 6-chlorothymol in 54% yield. This may be reduced by iron and alkali at 200° (G.P. 396,454; B., 1924, 825) to thymol. C. HOLLINS.

Production of santalol derivatives. S. G. S. DICKER. From VER. F. CHEM. IND. A.-G. (B.P. 278,982, 14.7.26).—*Santalyl bromoacetate*, prepared by treating santalol in chloroform solution with bromoacetyl bromide and quinoline, combines with hexamethylenetetramine to form a soluble, non-irritant compound with scarcely any bitter taste. The bromoacetate may be replaced by santalyl nitrate, sulphate, thiocyanate (prepared from santalyl chloride and sodium thiocyanate in dry acetone), or benzenesulphonate (prepared from santalol, benzenesulphonyl chloride, and pyridine in ether). C. HOLLINS.

Production of derivatives of organic amino- or imino-compounds. S. G. S. DICKER. From VER. F. CHEM. IND. A.-G. (B.P. 255,468, 14.7.26).—Amines, imines, amides, or imides (e.g., pyrrolidine, diethylbarbituric acid, or diaminodihydroxyarsenobenzene) are solubilised by condensation first with bromoacetyl bromide, then with hexamethylenetetramine. C. HOLLINS.

Preparation of monocyclic ketones, having more than nine ring members, and of their alkyl derivatives. M. NAEF and F. FIRMENICH (B.P. 263,153, 14.12.26. Switz., 15.12.25. Addn. to B.P. 235,540; B., 1925, 738).—Polymethylenedicarboxylic acids or their anhydrides are converted into cyclic ketones by heating with metals (or their oxides etc.) of the aluminium and titanium groups (excluding cerium and thorium) or with uranyl compounds. 3-Methylcyclopentadecanone, b.p. 125°/0.3 mm., is prepared by heating aluminium γ-methyltetradecane-αω-dicarboxylate in a vacuum at 300–500°, fractionating the distillate, and isolating the ketone as semicarbazone, m.p. 164°. 4-Methylcyclopentadecanone, b.p. 125°/0.3 mm. (semicarbazone, m.p. 166°), from δ-methyltetradecane-αω-dicarboxylic acid, and cyclononadecanone, m.p. 72°, b.p. 160°/0.3 mm. (semicarbazone, m.p. 184°), are similarly prepared. Erbium, yttrium, didymium, lanthanum, uranyl, titanium, zirconium salts, or mixtures of these may be used. The ketones have an odour of natural musk. C. HOLLINS.

Production of organic arseno-compounds. A. BINZ and C. RÄTH (B.P. 255,892, 22.7.26).—The formation of alkali-insoluble products in the reduction of hydroxylated organic arsinic acids, containing no amino-group, to arseno-compounds is prevented by using as reducing agent aldehyde- or ketone-sulphoxylic acids, e.g., formaldehydesulphoxylic acid, acetone-sulphoxylic acid, or diformaldehydesulphoxylic acid (hydroxymethyl hyposulphite; cf. Binz, Hueter, and Goldenzweig, A., 1918, i, 4; Binz, *ibid.*, i, 291). The freshly prepared acids may be used, or they may be introduced as sodium salts and acid added. Arseno-compounds are prepared from 2-hydroxypyridine-5-arsinic acid, its 3-bromo-derivative, and *o*-, *m*-, and *p*-carboxybenzenearsinic acids. C. HOLLINS.

Manufacture of 4-hydroxy-3-acetamidoaryl-arsinic acids. W. CARPMAEL. FROM I. G. FARBENIND. A.-G. (B.P. 278,789, 7.7.26).—A 4-nitro-2-acetamidophenol is reduced to amine and converted by Bart's method (B.P. 568 of 1911) into the corresponding 2-acetamidophenol-4-arsinic acid. 3-Acetamido-*o*-cresol-5-arsinic acid (B.P. 254,086) and 6-chloro-2-acetamidophenol-4-arsinic acid (B.P. 257,361; B., 1926, 932) have already been prepared by this method. 4-Nitro-2-acetamidophenol is reduced to the 4-amino-compound which is converted into 2-acetamidophenol-4-arsinic acid; 6-chloro-4-amino-2-acetamidophenol, m.p. 222—224°, gives 6-chloro-2-acetamidophenol-4-arsinic acid.

C. HOLLINS.

Production of soaps, cosmetics, and medicinal preparations containing succinic acid. H. H. VON KORNATZKI (B.P. 279,575, 7.8.26).—Rectified oil of amber and succinic acid, in the same relative proportions as they are obtained in the rectification with water of crude oil of amber, are added to the usual ingredients of soaps, cosmetics, and medical preparations.

S. S. WOOLF.

Removal of nicotine from tobacco products. C. PORT (B.P. 279,757, 16.7.27).—After subjecting the products to warm, moist air in a storage chamber for 2—6 days, they are transferred to a closed, uniformly heated vessel, the temperature of which is quickly raised to 150°. The generated steam is continually removed by means of a pump. H. ROYAL-DAWSON.

Purification of vaccines. F. BILLON (U.S.P. 1,649,381, 15.11.27. Appl., 2.6.26. Fr., 6.6.25).—The vaccine pulp is treated with a diluting mixture consisting of 1500 pts. of glycerin and 1 pt. of trichloronitromethane.

F. G. CROSSE.

Ampoules for the preparation of solutions, more particularly for medical and like purposes. "PHARMAGANS" PHARMACEUTISCHES INST. L. W. GANS A.-G. (B.P. 264,215, 22.7.26. Ger., 5.1.26).

Recovery of acetic acid (B.P. 268,778).—See V.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Latent image and development. H. J. VOGLER and W. CLARK (Brit. J. Phot., 1927, 74, 670—671).—The evidence for the view that developability of the latent image is occasioned by its having a certain minimum of size is summarised, and it is suggested that developability is due to the formation on the grains of a stable space-lattice of silver. Latent image centres may be incapable of acting as development centres if they do not form a lattice which is sufficiently stable to withstand the peptising tendency of the developer or, in the case of physical development after fixation, of the fixing bath.

W. CLARK.

Hypersensitisation and ultrasensitisation [of positive plates]. P. GUILLEMINOT (Bull. Soc. Franç. Phot., 1927, 69, 234—237).—Dyestuffs studied were Pinachrome, Pinaverdol, Pinaflavol, Pinacyanol, and Orthochrome of Meister Lucius, and Olochrome, Chlorochrome, and Erythrochrome of Calzavarra, the general

formula for hypersensitisation being distilled water 40 c.c., 90% alcohol 40 c.c., alcoholic dye solution (1/1000) 4 c.c. For ultrasensitisation, 10 c.c. of a 0.2% solution of ammoniacal silver chloride were added. Strict control of temperature is necessary otherwise fog results. Results showing the effects for exposure to various colours are tabulated for the positive plate "Lactate tons noirs." The basic dyes Pinaflavol and Olochrome confer practically no sensitivity by the methods used. Best results were obtained with Pinaverdol and Chlorochrome, which gave sensitivity values respectively 34—40 times the initial value. The red sensitisers Pinacyanol and Erythrochrome give good results in hypersensitisation, but bad fog when ammoniacal silver chloride is added to the bath. Blue sensitivity is reduced by the use of most of the dyes alone, an effect which is due to their screening action rather than to a true desensitisation. Addition of ammoniacal silver chloride does not change the screening effect but increases the sensitivity to the blue. The gamma of the plates is only slightly reduced by the treatment. It is possible to obtain fine-grained plates of high gamma with a speed higher than that of many negative plates.

W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

Safety explosives. P. NAOÛM (Z. angew. Chem., 1927, 40, 1351—1359).—An account is given, with special reference to German conditions, of the various stages by which freedom from explosions due to the use of explosives in dusty and fiery mines has been attained. The claim is made that any explosions at the present time can be attributed, not to the explosive, but to ignition of coal or firedamp by burning remnants of the detonator. The most recent German procedure for testing explosives prior to authorisation for use in mines is described. The problem of determining the duration and amount of flame following the initiation of an explosion is then discussed, and photographs are given of the flames from typical modern French and German explosives; it is deduced that, in this respect, the German explosives are in advance of the French. Examples are quoted to illustrate the loss in power due to the necessity of keeping the temperature of the explosion gases below a certain maximum.

S. BINNING.

Nitroglycerin washing process. A. SCHMID (Z. ges. Schiess- u. Sprengstoffw., 1927, 22, 308—311).—The loss of nitroglycerin occasioned by the normal washing method, consisting of three pre-washes (100 g. of oil to 40 c.c. of water at 30°, 35°, and 40° for 10 min.), a soda wash (40 c.c. of 4% soda at 40° for 30 min.), and a final water wash (40 c.c. at 30° for 10 min.), is only about 1% (calculated on the glycerin used), the alkaline wash being responsible for at most 0.2% loss. Stabilisation is slow owing to the insolubility of soda in nitroglycerin, and may be hastened by the use of aqueous ammonia as the wash liquor or by adding to the soda solution a small quantity of an amine, e.g., aniline, which dissolves readily in the nitroglycerin, forms with the free acid a water-soluble salt, and is regenerated when this salt passes into the aqueous alkaline phase. The amine should not be added until about 2 min. after the beginning of the wash, when the bulk of the free acid has been

neutralised (as otherwise the oil becomes discoloured), and stirring continued for 3–5 min. A single wash of this nature yields a product of high heat-test and a final water wash is unnecessary. The amine remains dissolved in the nitroglycerin at the end of the wash, but it acts as a stabiliser and, owing to the small amount present, the explosive properties of the product are not affected. An apparatus suitable for a continuous washing of nitroglycerin by this process is described.

W. J. POWELL.

Nitrocelluloses soluble in alcohol. S. K. HAGEN (*Z. angew. Chem.*, 1927, 40, 1359–1361).—The solubilities in ethyl alcohol have been investigated for nitrocelluloses prepared from cotton that has been heated at $155^{\circ} \pm 2^{\circ}$ in a stream of nitrogen for various periods. Experiments were also made on nitrocelluloses prepared from mercerised cotton and from cotton that had been both heated and mercerised. The percentage of nitrogen was lowered to some extent by these treatments, which, however, had a marked effect on the solubility in alcohol. Nitrocellulose from untreated cotton had a solubility of 28%, which was doubled when the cotton had been heated for 6 hrs. Complete solubility in alcohol was not attained even after prolonged heating. Mercerisation, although more effective than a 48 hrs. heating, failed to yield a completely soluble nitrocellulose, but a cotton that had been both mercerised and heated yielded on nitration a nitrocellulose which had a solubility of 97–99% in alcohol.

S. BINNING.

PATENTS.

Manufacture of explosives. R. K. HEZLET and A. HIGHFIELD (B.P. 279,529, 25.6.26).—The gelatinisation of nitrocellulose by nitroglycerin or other nitric esters is facilitated by using a gelatiniser which contains suitable proportions of polar and non-polar groups. A mixture of an alcohol and/or nitroglycerin or other nitric ester is suitable. In the cases of blasting gelatin, gelignite, or gelatin dynamite the alcohol is added to a mixture of nitroglycerin and nitrocellulose. In manufacturing ballistite small amounts of alcohol are added during the rolling of the paste. The process is applicable to both solvent and non-solvent powders. The surface of granular powders may be gelatinised by spraying the grains with alcohol or with an alcoholic solution of nitric esters.

S. BINNING.

Propellant powder. C. H. KECK (U.S.P. 1,649,784, 15.11.27. Appl., 6.11.26).—The powder contains nitrocellulose 74.5%, dinitrobenzene 20%, mononitroxylene 5%, and a stabiliser 0.5%.

S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Determination of oxygen in drinking and river water. L. W. HAASE (*Gas- u. Wasserfach*, 1927, 70, 1065–1067).—In preparing the precipitate of manganous hydroxide in Winkler's method of determining oxygen in water, the sodium hydroxide should not be added before the manganous chloride, as this results in a visible liberation of oxygen which persists for several days. Errors are liable to occur on account of the heat evolution, with subsequent contraction of the liquid,

accompanying the formation of manganous hydroxide, and to differences in temperature between the water sample and the surrounding air. Care must be exercised in removing the clear liquid above the hydroxide precipitate, to prevent any unoxidised hydroxide coming into contact with the atmosphere. To destroy traces of nitrites in water a few drops of 5% sodium azide solution should be added before acidification, and to ensure complete liberation of iodine after acidification the mixture must be kept in a closed flask for at least 10 min. before titrating with 0.01N-thiosulphate. The latter must be standardised with potassium chromate shortly before each determination. In taking the original water sample, it is important to avoid the passage of air bubbles through the water.

W. T. K. BRAUNHOLTZ.

Determination of dissolved oxygen in water. V. SUBRAHMANYAN (*J. Agric. Sci.*, 1927, 17, 468–476).—A modification of Thresh's method (B., 1890, 327) is described, which eliminates the error due to loss of iodine, carried away by the gas passing through the apparatus during the titration. The method also obviates the need for making separate determinations of the dissolved oxygen in the reagents used.

C. T. GIMMINGHAM.

Determination of free chlorine in drinking and washing water. W. OLSZEWSKI and H. RADESTOCK (*Pharm. Zentr.*, 1927, 68, 733–735).—The limits of sensitiveness and methods of preparing the appropriate solutions are given for the determinations by means of benzidine and potassium iodide, respectively. The former is positive for concentrations of free chlorine between 0.03 and 0.1 g. per litre; for higher concentrations the iodide method is more suitable. The temporary hardness of the water to be tested must be known before the benzidine method can be employed; tables are given of the quantities of reagent and acid to be employed with increasing hardness.

S. I. LEVY.

Volumetric determination of sulphates in water by Bahrdr's method. M. KEHREN and H. STOMMEL (*Chem.-Ztg.*, 1927, 51, 913–915, 934–935).—A series of tests on the volumetric method of determining sulphates in water by filtration through permutit followed by titration with potassium palmitate (cf. Bahrdr, B., 1927, 238) has shown that for hard waters at least 100 g. of permutit must be used, and the rate of filtration should not exceed about 400 c.c./hr. For the regeneration of the permutit a 10% solution of sodium chloride should be used. Iron compounds in the water interfere so that ferruginous water must first be filtered through manganese permutit.

A. R. POWELL.

Contact insecticides. TATTERSFIELD and GIMMINGHAM.—See XVI.

PATENTS.

Stable dry mixtures of soluble colloidal metals or metalloids with indifferent materials. F. STEINITZER and P. LOOCK (G.P. 443,512, 28.9.24).—Insecticide powders comprise a mixture of a soluble metal colloid and a purified and dehydrated, inert material such as talc, kieselguhr, or barytes.

A. R. POWELL.

Fumigant. R. C. ROARK (U.S.P. 1,649,254, 15.11.27. Appl., 4.8.24).—See B.P. 234,456; B., 1926, 564.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JANUARY 20, 1928.

I.—GENERAL; PLANT; MACHINERY.

Porosity and permeability of porous bodies.

W. L. HOWE and C. J. HUDSON (J. Amer. Ceram. Soc., 1927, 10, 443—448).—The apparent porosity, which is measured by means of an air-expansion porosimeter, is expressed as the percentage of the volume occupied by pores, whilst permeability is defined as the quantity of air flowing through an article of definite area and thickness in unit time, when tested under an arbitrary constant pressure difference. In measuring the permeability, air passes from a pressure-equalising tank to a plate box, where a pressure of 2 in. in excess of atmospheric is allowed to build up under the experimental plate (12 in. \times 12 in. \times 1 in.). Under these conditions the flow of air through the plate is determined by means of a previously calibrated gauge. An air-seal confines the passage of the air to the plate under investigation. The plates were made of a granular aluminous material of known grain size bonded with clay and fired at cone 12. The permeability was varied over a wide range, without appreciably changing the apparent porosity, by the adequate selection of grit size. The finer the grit the lower is the permeability. The permeability can also be changed by varying the content of bond clay, in which case the apparent porosity is changed. High bond contents result in producing bodies of lower apparent porosities and permeabilities. The permeability increases with increasing grain size. As finer grains are employed the body appears to become more dense; actually, however, it contains a greater number of finer pores, practically maintaining constant porosity.

A. T. GREEN.

Absorption towers. KAUFFMAN.—See II.

PATENTS.

Furnaces or kilns for heating goods. BRIT.

FURNACES, LTD., and E. W. SMITH. From SURFACE COMBUSTION Co. (B.P. 279,317, 22.3.27).—A furnace for, e.g., reheating steel is constructed with two gas producers, two recuperators, and two heating chambers preferably built into one structure. One heating chamber may be square-shaped to contain sheets, and the other elongated to contain bars. One producer is normally connected to one heating chamber and the other to the other, but there is provision for the gas from both producers to mingle and pass to either chamber, similarly with the heated secondary air from the recuperators.

B. M. VENABLES.

Annular rotary-hearth (sole plate) ovens. TROCK-

NUNGS-, VERSCHWELUNGS-, U. VERGASUNGS-GES.M.B.H., and F. BARTLING (B.P. 279,343—4, 26.5.27).—(A) The hearth which carries the goods is heated from below by stationary surface-combustion burners. (B) In addition

to the usual sand seal, an oven which has its rotary hearth heated from below is provided with a water-seal and cooling device to prevent escape of heat and to ensure that the supporting and driving mechanism remains cool.

B. M. VENABLES.

Heat interchangers. A. E. LEEK (B.P. 279,663, 8.12.26).—A heat exchanger with pairs of concentric gas and water tubes, in which the area available for the passage of air and/or gas is increased in steps from the inlet to the outlet.

B. M. VENABLES.

Producing a mixture of air and combustion products to be used for combustion in furnaces provided with rotating air preheaters. AKTIEBOLAGET LJUNGSTRÖMS ÅNGTURBIN (B.P. 263,492, 20.12.26. Swed., 22.12.25).—In addition to interchange of heat by conduction without mixing, a portion of the flue gases exhausting from the preheater is mixed with the entering air, whereby the primary combustion air will contain a proportion of carbon dioxide, which, by reduction to monoxide, will keep the lowermost layers of fuel and the firebars comparatively cool, avoiding corrosion and slagging of ash.

B. M. VENABLES.

Heating apparatus. F. PUENING (B.P. 255,866, 19.7.26. U.S., 24.7.25).—In a heating apparatus where the hot gases are pulsated by a piston, the latter is kept cool by air (or gas) admitted to its interior and exhausted through perforations on its surface in such a manner that a layer of cool air is formed, which is continually renewed and gradually mixes with the burning gases. The piston rod may be surrounded by an auxiliary piston and cylinder to pump the air.

B. M. VENABLES.

Apparatus for calcining cement, lime, dolomite, magnesite, etc., and for agglomerating ores. E. W. STOLL (G.P. 444,569, 12.9.24).—The finely-divided material, gaseous, liquid, or pulverised solid fuel, and preheated combustion air are injected downwards or in a horizontal direction into a combustion chamber below a steam boiler, and the product is withdrawn at the bottom between cooling tubes.

L. A. COLES.

Pilger mills. SCHLOEMANN A.-G. (B.P. 279,353, 13.6.27. Ger., 18.3.27).—A feeding device for a pilger mill is provided with a brake comprising a quantity of water on one side of the feed piston which compresses air into a closed vessel during the feed motion, the pressure adverse to the feed rising rapidly and forming a practically definite but shockless stop, the position of which can be varied by altering the amount of air trapped in the vessel.

B. M. VENABLES.

Crusher. ALLIS-CHALMERS MANUF. Co., and R. C. NEWHOUSE (B.P. 279,742, 13.6.27).—A gyratory crusher which is driven by a motor, above, through a high-speed

shaft within the hollow crusher shaft, to reducing gearing and eccentric, below.

B. M. VENABLES.

Centrifugal machine. CONSTRUCTION FRANÇ. D'APPAREILS DE LAITERIE, and A. MINET (B.P. 279,318, 22.3.27).—In a centrifugal apparatus of the peg-top type, at speeds below the critical, the spindle is supported by centrifugal weights attached to it which slide against a fixed bearing surface, but at higher speeds the weights lift from the fixed surface, and the spindle and bowl run unsupported except at the bottom. B. M. VENABLES.

Concentrator. A. H. STEBBINS (U.S.P. 1,648,285—6, 8.11.27. Appl., [A] 2.7.26, [B] 19.7.26).—(A) A shaking table on which concentration is effected by air which is blown through the material in channels on the surface. (B) A shaking table; no mention of air or water is made in the principal claim.

B. M. VENABLES.

Classifying or separating machine. P. H. RAUN, ASSR. to FREDERICK IRON & STEEL CO. (U.S.P. 1,647,812, 1.11.27. Appl., 7.1.26).—A separating machine comprises a number of rollers adjustably spaced in the same inclined plane, and having at the lower ends apertures through which water is sprayed to remove material lodging between them. Water sprays external to the rollers are also provided.

T. S. WHEELER.

Separating or classifying machine. W. A. RIDDELL (U.S.P. 1,647,815—6, 1.11.27. Appl., [A] 2.2.26, [B] 9.3.26).—(A) A separating machine comprising a number of rollers adjustably spaced in the same inclined plane is fitted with discs rotating between the rollers to prevent material lodging between them. (B) The rollers belong alternately to one of two sets, and means are provided for moving one set vertically with respect to the other to vary the distances between adjacent rollers.

T. S. WHEELER.

Apparatus for the treatment of materials by froth flotation. ELEKTRO-OSMOSE A.-G. (GRAF SCHWERIN Ges.) (G.P. 443,616, 22.4.25).—To prevent adhesion of the froth to the side walls of the flotation vessel, secondary walls comprising a series of movable plates are provided within the froth zone. By means of a suitable mechanism, these walls may be drawn forwards in the direction of flow of the froth to accelerate removal of the froth from the flotation vessel.

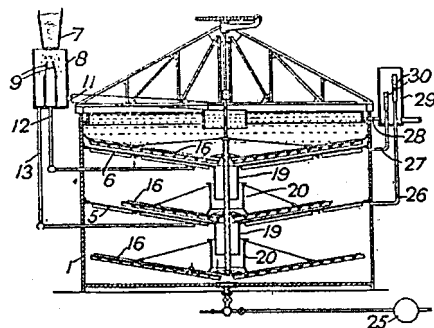
A. R. POWELL.

Apparatus for mixing and proportioning [liquid and solid] materials. E. P. HALLIBURTON (U.S.P. 1,649,062, 15.11.27. Appl., 16.9.26).—A feed hopper delivers the solid materials into an outlet pipe which is provided with a number of jets of the liquid. Each jet is supplied by its own pump, and the pumps are mechanically connected so that the ratio of their deliveries is positively determined.

B. M. VENABLES.

Separation of solids from liquids [in thickeners]. DORR Co., Assees. of W. C. WEBER (B.P. 262,479, 6.12.26. U.S., 5.12.25).—In a multi-deck or tray thickener, all the settled thick pulps are discharged at one place, viz., the extreme bottom, and dilution of the thick pulps by thin pulp is prevented. In the figure, 7, 8, and 9 indicate a feed splitter which ensures an equal supply of thin pulp to each of three settling compartments. The settled pulp from an upper compartment falls through a stationary sleeve or boot, 19, which is surrounded by

another sleeve or boot, 20, rotating with the rakes, and mingles with the settled pulp collected in the next lower compartment and so on until the lowest level of all is reached, and the combined thick pulp is removed through a pipe by, e.g., a diaphragm pump, 25. The



dilution of the thick pulp by thin feed pulp on its passage downwards is prevented by the sleeves, 19, extending below the sludge level of the lower compartment and by the adjustment described below. The clear overflow is drawn off through pipes 26, 27, and 28 from the top-most part of each compartment, and the hydrostatic head of the clear liquid in 26 and 27, is balanced against the head of the heavier thick pulp in the central column, and by altering this head by means of adjustable sleeves, 30, on the overflow ends of pipes 26 and 27, the rate at which the thick pulp is allowed to fall from each tray may be adjusted, and once adjusted the balance of thick pulp against clear liquid will be automatic, so that thin pulp cannot short-circuit through the central column.

B. M. VENABLES.

Separation of solids or semi-solids from liquids by drying or concentrating. J. A. REAVELL (B.P. 279,544, 27.7.26).—Heated air (or other gas) passes downwards with a whirling motion through a cylindrical casing at the top centre of which the liquid is sprayed centrifugally and horizontally. The liquid spray may be surrounded by a current of cold air which prevents premature evaporation. On arrival near the bottom of the apparatus the air is caused to turn sharply upwards into a zone of still larger diameter, so that its speed is reduced to such an extent that practically all the dried powder is deposited into the hopper-shaped bottom of the vessel.

B. M. VENABLES.

Washing solid material in centrifugal apparatus. F. SANDER, and I. G. FARBERIND. A.-G. (G.P. 444,062, 31.3.25).—The tubes conveying the washing water are spaced at such a distance on the circumference of a centrifugal drum that the contents can be discharged outwards. The orifices of the tubes are covered with filtering material.

L. A. COLES.

Drying apparatus with grate firing. HERRMANN & SÖHNE, KOMM.-GES. (G.P. 444,572, 23.8.23).—The drying chamber is protected from overheating by the provision of a lattice work of refractory bricks, oblong in shape and tapering upwards, between the grate and the walls of the chamber.

L. A. COLES.

Drying of moist material. KOHLENVEREDLUNG A.-G. (G.P. 443,272, 19.11.22).—The material is dried

in one or more compartments heated externally by steam at a temperature above that of the hottest of the escaping vapours, and drying is completed by replacing the steam by furnace gases. L. A. COLES.

Apparatus for mixing, compacting, degassing, or grinding more or less viscid material. A. SONSTHAGEN and G. PÖVERUD (B.P. 279,707, 6.4.27).—A mixer for compacting, degassing, or grinding viscous material is constructed with beaters giving an ordinary stirring motion and with shoes sliding on a surface which produce a wiping or grinding action. The grinding surfaces are provided with grooves, which ensure that there is always a film of material present and also act as vents for any gases emitted. Propellers are provided to keep the material in circulation between the two zones and a heating jacket may be provided.

B. M. VENABLES.

Atomiser. A./S. NIRO (B.P. 259,922, 4.8.26. Denm., 13.10.25).—A centrifugal atomiser comprising radial blades attached to a lower rotating disc is also provided with an upper disc either attached to the edges of the vanes or stationary, to prevent air being drawn in with the liquid.

B. M. VENABLES.

Atomisation of fluids. J. Y. JOHNSON. From INDUSTRIAL ASSOCIATES INC. (B.P. 279,925, 5.2.27).—A centrifugal sprayer comprises a centrifugal bowl to which the liquid is admitted, and in which it remains long enough to be accelerated to the full speed of the bowl. The spray is formed by the overflow of the liquid at both ends of the bowl.

B. M. VENABLES.

Spray-drying apparatus. W. H. DICKERSON, Assr. to INDUSTRIAL WASTE PRODUCTS CORP. (U.S.P. 1,648,939, 15.11.27. Appl., 9.1.23).—A stream of heated drying gas passes downwards without whirling through a cylindrical container, the liquid to be evaporated is sprayed by centrifugal means across the gas stream, and at a considerable distance below the spray cool gas is admitted to mix with the down-flowing combined stream. A collector is provided for dry material, and a wet one for vapours and any residual solid material, and the flow is induced by a fan.

B. M. VENABLES.

Vertical evaporator with long upright tubes. Soc. ANON. APPAREILS ET ÉVAPORATEURS KESTNER (G.P. 443,510, 5.7.24. Fr., 25.7.23).—The tubes are arranged so that channels for distributing the liquid in the lower compartment are formed between them, and the liquid from the centre or from the circumference of the compartment can flow through not more than three or four of the sets of tubes. Horizontal distributing plates are attached to opposite sections of the outer walls of the apparatus.

L. A. COLES.

Evaporator with horizontal tubes. E. SCHLEGEL (G.P. 443,740, 15.6.24).—The tubes of each group communicate with one another through compartments in front closed by a common cover, the removal of which exposes the interiors of all the tubes. The liquid from one group of tubes flows into another group through valves regulated by floats or by hand from the exterior of the apparatus.

L. A. COLES.

Apparatus for evaporating (concentrating) liquids without employing a vacuum. A. WURM (G.P. 443,511,

19.12.24. Addn. to G.P. 354,860; B., 1922, 737 A).—Means are described for improving the spraying effect of the bucket wheels in the apparatus described previously.

L. A. COLES.

Apparatus for rendering liquids homogeneous. MASKINFABR. "RAMIJE" (Dutch P. 16,212, 3.8.25).—The liquid flows through a channel on to the surface of a rotating cone enclosed in a suitable casing, and is forced between the cone and the casing.

L. A. COLES.

Clarification of liquids and solutions. E. MERCK, CHEM. FABR., Assecs. of P. LANGENKAMP (G.P. 443,567, 3.9.25).—A powdered mixture of equivalent weights of potassium ferrocyanide and a compound which, after dissolution, forms an insoluble precipitate with it, such as copper sulphate, is added to the liquid or solution.

L. A. COLES.

Manufacture of absorbent materials. BRIT. DYE-STUFFS CORP., LTD., J. BADDILEY, and E. CHAPMAN (B.P. 280,262, 15.7.26).—Cotton wadding, absorbent paper, kieselguhr, or other materials intended for the absorption of aqueous liquids are impregnated with a wetting-out agent, viz., sulphonated aromatic, hydro-aromatic, aromatic-aliphatic, or polynuclear hydrocarbons, sulphonated alkylated mineral oil fractions, etc. or their salts, and dried. The rate of absorption by the treated materials is increased.

C. HOLLINS.

Liquid flow and viscosity meters. A. BERAUD and G. JACQUET (B.P. 271,846, 4.5.27. Fr., 27.5.26).—The apparatus comprises an arc-shaped piston, counter-balanced and spring-controlled to swing about a pivot and provided with a pointer moving over a scale. The piston works with considerable clearance in an arc-shaped "cylinder," and the liquid on its way to the point of consumption is forced through the clearance space, the drag deflecting the pointer according to the rate of flow.

B. M. VENABLES.

Manufacture of plates or walls of vessels to be heated or cooled by passage of fluid through tubes. R. SAMESREUTHER (B.P. 273,306, 22.6.27. Ger., 22.6.26).—Instead of the known construction of drawn metal tubes cast in the cast-metal walls of a vessel, which is heavy and mechanically weak, a strong light vessel of wrought plate (e.g., steel) may have the tube coil welded on to it.

B. M. VENABLES.

Heating of gases and vapours. H. DUPUY (B.P. 272,886, 31.5.27. Fr., 18.6.26).—A mass of solid combustible matter has its temperature raised by passing a combustion-supporting gas through it, the gas to be heated is then passed through, and so on in turn. Means for admitting a small quantity of igniting combustible are provided should the temperature drop too low for spontaneous re-ignition.

B. M. VENABLES.

Transmission of heat from one gas to another. GES. F. LINDE'S EISMASCHINEN A.-G. (B.P. 256,271, 3.8.26. Ger., 31.7.25).—One gas transfers its heat (or cold) to a circulating liquid by direct contact, and the liquid transfers the heat (or cold) to the other gas. For example, in the liquefaction of ethylene using toluene as a heat carrier, the ethylene gas under high pressure at ordinary or cooling-water temperature flows counter-current to cold toluene under high pressure in one tower,

the liquid being raised to ordinary temperature and the gas is thereby chilled sufficiently to produce partial liquefaction on expansion. The unliquefied ethylene at lower pressure meets the toluene in another tower and cools it. If a single liquid with the necessary properties—f.p. below the lowest temperature attained and substantially non-volatile at the highest temperature—is not obtainable, the heat-transfer may be in more than one stage, using a different liquid in each stage.

B. M. VENABLES.

Pyrometers. SIEMENS & HALSKE A.-G., Assees. of VER. DEUTS. EISENHÜTTENLEUTE (B.P. 274,096, 7.7.27. Ger., 8.7.26).—In a pyrometer in which a portion of the gas of which the temperature is to be measured is drawn through a conduit surrounding the thermometer bulb or thermocouple, the latter is surrounded by bodies which, having good heat conductivity and large area, quickly attain the temperature of the gas and protect the pyrometer from radiation effects from the wall of the conduit.

B. M. VENABLES.

[Means for measuring the extent of the reaction in] apparatus for effecting chemical reactions in liquid media, or for concentrating liquids. I. G. FARBENIND. A.-G., Assees. of M. LUTHER and E. HOCHHEIM (G.P. 443,454, 17.12.25).—The extent to which the reaction has taken place is ascertained by measuring the mechanical resistance of the liquid to a stirrer rotating in it, *e.g.*, by observing the distortion of a spring connecting the two halves of the main shaft carrying the blades. The method is useful, *e.g.*, in the production of condensation products from carbamide and formaldehyde.

L. A. COLES.

Mechanically operated gas analysers. G. H. BARKER. From A. B. CUNNINGHAM (B.P. 279,319, 24.3.27. Cf. U.S.P. 1,311,952; B., 1920, 708).—A measured quantity of gas is taken, subjected to absorption of one of its constituents, and remeasured. The measuring vessel and mechanism are submerged in non-volatile oil, and the operations are performed automatically.

B. M. VENABLES.

Determination of the absolute moisture content of air and other gases. L. LÖWENSTEIN (G.P. 444,760, 29.6.24).—Air or gas is brought to a constant temperature before entry into the hygrometer, by heating it above the desired temperature and then passing it through a coil immersed in a liquid which is kept boiling and has a constant b.p. below the temperature of the gas.

L. A. COLES.

Carrying-on of [exothermic] catalytic reactions. J. M. SELDEN (U.S.P. 1,647,317, 1.11.27. Appl., 2.1.20).—The ducts containing the catalyst are immersed in a bath of molten metal, cooled externally by a current of air the velocity of which is thermostatically controlled.

T. S. WHEELER.

Pulverising and separating machinery. J. C. CARLINE (U.S.P. 1,651,372, 6.12.27. Appl., 19.7.26).—See B.P. 265,841; B., 1927, 287.

Regenerative heat-exchange device. F. LJUNGSTRÖM, Assr. to AKTIEBOLAGET LJUNGSTRÖMS ÅNGTURBIN (U.S.P. 1,652,025, 6.12.25. Appl., 11.4.21. Swed., 23.4.20).—See B.P. 162,250; B., 1922, 795.

Fire extinguisher. J. L. FOHLEN (U.S.P. 1,650,985, 13.10.23. Ger., 7.10.22).—See E.P. 205,110; B., 1924, 497.

II—FUEL; GAS; TAR; MINERAL OILS.

Rational utilisation of Rumanian lignites. II. Attempts to briquette the semi-coke. I. BLUM (Bul. Chim. Soc. Română Stiin., 1926, 29, 3—13; cf. B., 1926, 652).—Semi-cokes prepared by carbonising Rumanian lignites in a rotary retort at 500° have been crushed and briquetted, using as binder 6—7% of a mixture of lignite pitch and petroleum pitch. By distillation and partial oxidation about 45% of the lignite tar could be converted into pitch for this purpose. The briquettes are superior in heating qualities to the lignite itself or to the semi-coke, and are capable of withstanding transport.

A. B. MANNING.

Auto-ignition temperatures. H. J. MASSON and W. F. HAMILTON (Ind. Eng. Chem., 1927, 19, 1335—1338).—If drops of a combustible liquid fall on to a surface, the temperature of which is gradually raised, a minimum temperature is eventually reached at which a drop ignites immediately after contact with the surface; this is termed the auto-ignition or spontaneous ignition point. Its actual value depends on a number of variables which in previous experiments have been imperfectly controlled. In the apparatus described all variables except the composition of the liquid and the temperature of the surface are kept constant. The surface consists of a platinum-platinum-rhodium thermocouple welded to a platinum plate which is heated in an electric furnace. The liquid is dropped from a hypodermic needle, and dry air can be passed over the hot plate as required; by raising the temperature sufficiently slowly the ignition point can be determined and repeated to within 1° F. Figures for a large number of pure organic compounds are given and possible interpretations of these are discussed.

R. H. GRIFFITH.

Determination of hydrogen in fire-damp. HEYER (Kali, 1927, 21, 147—148; Chem. Zentr., 1927, II, 310—311).—A colorimetric modification of von Zenghelis' method (cf. B., 1910, 1376) is described. The gas, after removal of carbon monoxide and hydrogen sulphide, is passed through a suitable quantity of a solution prepared by dissolving 0.1 g. of molybdenum trioxide in dilute sodium hydroxide solution, adding 0.1 g. of colloidal platinum mixed with sodium protalbin-ate, and diluting to 200 c.c., the solution being acidified with dilute sulphuric acid immediately before use. The solution is compared with standards prepared by treating further quantities with gas having a known content of hydrogen.

L. A. COLES.

Fractionation analyses of several fuel gases with special reference to illuminants. W. P. YANT and F. E. FREY (Ind. Eng. Chem., 1927, 19, 1358—1361).—A number of gases of high calorific value have been analysed in detail with respect to the saturated and unsaturated hydrocarbons present. Gas from low-temperature carbonisation under four different sets of conditions, coke-oven gas, carburetted water-gas from coke and anthracite, oil-gas, and mixed coal- and water-

gas were examined. The sample was separated by distillation into fractions containing 1, 2, 3, 4, and more than 4 carbon atoms per molecule, and the proportion of unsaturated hydrocarbons was determined by absorption in sulphuric acid. Comparison of the analyses of gases produced by increasing carbonisation temperatures show the degradation of the larger molecules; the effect of varying temperature and hydrogen concentration on the ratio of olefine to olefine plus paraffin is demonstrated. Orsat analyses were carried out on the gases in order to compare their accuracy with that of the fractionation method. Calorific values for individual olefines are given which may be used for calculating the value of mixtures. R. H. GRIFFITH.

Manufacture of water-gas of low specific gravity. L. STEIN and L. J. WILLIEW (Chem. Met. Eng., 1927, 34, 676—677).—Straight coal-gas has d 0.38—0.43, whereas ordinary carburetted water-gas has d 0.70. The supply of the latter at periods of maximum demand leads to difficulties owing to great variations in the density of the gas as it reaches the consumer. It was found, however, that the hydrogen content of carburetted water-gas could be substantially increased by spraying oil into the superheater during the "back-run," the hydrogen being derived from the over-cracking of the oil. By this method of operation an additional 0.5 gal. of oil was used per 1000 cub. ft. of gas, with a saving of 7 lb. of coke. The gas thus made had d 0.50—0.55, and the effective capacity of the water-gas generator was increased 25—50%. The above refers to gas of 550 B.Th.U. throughout. C. IRWIN.

Distillation of Schwabian shale by means of inert waste gases. K. NEUBRONNER (Petroleum, 1927, 23, 1567—1572).—On account of the low content of bituminous substances in Schwabian shale, distillation for oil or gas alone does not pay, and economic operation of the industry depends on use of the ash for the preparation of cement etc. Like other bituminous clay marls, this shale shows a tendency to sinter or even melt at fairly low temperatures, and on this account careful control of carbonisation is necessary. A two-stage process is described in which the oil is first distilled, the shale being heated internally by a suitable mixture of waste gas and air. After this the ash is burned completely in another vessel, great care being necessary at this stage to prevent clinker formation. Operating data, such as throughput, oil and gas yields, etc., are given for different shales, and heat balances for carbonisation in the laboratory are compared with those obtained on the large scale. R. H. GRIFFITH.

Composition and distillation of shale present in potash mines. P. DOMEK (Bull. Soc. Ind. Mulhouse, 1927, 93, 462—467).—The shale consists mainly of inorganic constituents, but contains 7.13% of organic matter, and when distilled at 400—500° in closed retorts it yields a gas consisting chiefly of carbon dioxide and methane, an ammoniacal liquid (d 1.004), and an oily product (d 0.854). Details are given of the results obtained in fractionally distilling these products. A. J. HALL.

Dry refining of Rumanian oils. P. NOBEL (Petroleum, 1927, 23, 1399—1401).—The presence of naph-

thenic acids in Rumanian petroleum has made difficult the adoption of dry-refining methods, as these substances or their sulphonation products are not completely removed by fuller's earth, and the oils darken quickly. Older processes of washing with aqueous alkali gave troubles with emulsification, but a new method of alkali treatment has proved successful. Oxides, carbonates, etc. of the alkali metals or alkaline-earths are added to the crude oil, either in the still or in a suitably adapted preheater, and the mixture is agitated for some hours before distillation. The naphthenic acids remain in the still in the form of soaps; when lime is added the resulting asphalt has a higher ash content and is somewhat more brittle than a normal sample of the same hardness; if caustic soda is used the softer asphalts are gummy and emulsify easily with water. The distillate is almost completely neutral and suitable for dry refining. The general advantages of the method are emphasised with heavy oils which normally show high refining losses. R. H. GRIFFITH.

Determination of nitrogen bases in petroleum oils. R. H. MCKEE and H. H. PARKER (Ind. Eng. Chem., 1927, 19, 1343—1344).—When sulphuric acid absorption is used to determine the unsaturated content of an oil, nitrogen bases are simultaneously removed. A number of artificial mixtures containing basic substances have now been treated with sulphuric, hydrochloric, and acetic acids of varying strengths in Babcock test bottles whirled in a centrifuge. From the results obtained it is demonstrated that 25% acetic acid is most generally suitable for removing bases from a petroleum distillate, although hydrochloric acid could be employed at varying concentrations in different cases. R. H. GRIFFITH.

Determination of volatility of gasoline. R. STEVENSON and J. A. BABOR (Ind. Eng. Chem., 1927, 19, 1361—1366).—The Engler distillation method for the determination of the volatility of a gasoline is unsatisfactory because the process of vaporisation in an automobile engine is not a fractionation. The use of the dew point as an indication of the temperature of complete vaporisation has been recently introduced, and the principle is now developed to apply not only to gasoline but to gasoline-air mixtures. The apparatus consists of a vaporiser, jacketed with superheated steam, in which the gasoline is volatilised, and a Dewar bulb containing the black-surface dew indicator which is under intense illumination. The indicator is cooled at a suitable speed by an internal stream of compressed air. Results are compared favourably with those given by the phase-change method, and, for pure substances, with their b.p. Modifications of the apparatus necessitated by its use for gasoline-air mixtures are described, but in practice these values need seldom be determined, as an empirical relation between the equilibrium end-point and the dew points of mixtures with air has been found. R. H. GRIFFITH.

Emulsification of transformer oils. G. INCZE (Petroleum, 1927, 23, 1398—1399).—The breakdown voltage of an oil depends on its purity, and is lowered by the presence of mechanically suspended impurities, air bubbles, and moisture. Water may be present by absorption from the air or by decomposition of the oil during use, and tables are given showing the lowering of

the breakdown voltage caused by small amounts of water. A method of testing the suitability of an oil is described which depends on the ease of emulsification with water; the test also indicates the degree of efficiency to which the oil has been refined.

R. H. GRIFFITH.

Efficient design and operation of absorption towers [for gasoline]. H. L. KAUFFMAN (Chem. Met. Eng., 1927, 34, 671—672).—A bubbling tower used for the extraction of petrol from natural gas consists of ten trays with 1½-in. seals and a "mist extractor" with a 6-in. seal. The lower trays may be fitted with coils for temperature control. Such a tower 30 ft. × 6 ft. has a capacity of 8—12 million cub. ft. of gas per 24 hrs., at 30 lb. working pressure. The bubble caps occupy practically the whole area of the trays, and are covered by a 1-in. layer of mesh. Absorption oil should be of good colour, low viscosity, sufficiently low vapour pressure, and low in unsaturated compounds.

C. IRWIN.

PATENTS.

Apparatus for extracting volatile matter [from coal]. K. E. CLAYTON-KENNEDY, Assr. to AMER. HYDROCARBON Co., INC. (U.S.P. 1,647,273, 1.11.27. Appl., 26.5.26).—A tumbler for use in rotary kilns for the distillation of coal is formed of two rectangular metal sheets with staggered perforations, intersecting at right angles and somewhat shorter in length than the kiln.

T. S. WHEELER.

Apparatus for carbonising wood. R. MALBAY (F.P. 619,791, 3.8.26).—A number of retorts are grouped in pairs coupled together at the top by a tube leading into a suction chamber, and by another tube leading into a second chamber. A fan is provided to circulate the gases through both retorts.

L. A. COLES.

Apparatus for indicating accumulations of fire-damp in mines, and for preventing explosions therefrom. F. W. MOLL SÖHNE (G.P. 443,270, 30.6.25).—A portion of the air surrounding a methane indicator is drawn through the lamp in such a manner that the temperature of the flame varies with the methane content of the air. When the temperature exceeds a predetermined limit, it closes a weak electric circuit, and the supply of air to that portion of the mine is regulated automatically or by hand, so as to minimise risk of explosion.

L. A. COLES.

Apparatus for indicating the methane content of the air in mines. H. FLEISSNER (G.P. 443,269, 13.9.25).—A benzine safety lamp contains a vertical sheet of incandescent material in the form of gauze or grating placed immediately above the normal height of the flame. An increase in the methane content of the air is indicated by extension of the incandescent area.

L. A. COLES.

Drying and removing oil from gases and vapours. G. DE GEYTER (F.P. 620,874, 30.8.26. Belg., 5.5.26).—The gases pass over rotating conducting surfaces in a cylindrical vessel provided with consecutive inlets and outlets for the gases.

L. A. COLES.

Refining heavy mineral oils. L. EDELEANU, K. PFEIFFER, K. GRESS, and P. JOBECK (U.S.P. 1,651,328, 29.11.27. Appl., 13.9.24. Ger., 19.9.23).—The oil is mixed with liquefied sulphurous acid and the mixture

passes to a settling chamber, the velocity of the liquid entering this chamber being retarded. Separation of the oil from impurities and sulphurous acid is effected by the action of gravity.

C. O. HARVEY.

Treatment of lubricating oil stocks. J. W. WEIR and W. J. RYAN, JUN. (U.S.P. 1,650,782, 29.11.27. Appl., 3.3.26).—Substantially sludge-free, sulphuric acid-treated lubricating oil stock is treated with an alkaline solution in such quantity that practically no excess of alkali above that required for neutralisation remains, and the soap is removed from the resulting emulsion by treatment at a temperature above 100° with a finely-divided adsorbent material and subsequent filtration.

C. O. HARVEY.

Oil-distillation process. E. C. D'YARMETT, Assr. to O. K. HERNDON (U.S.P. 1,646,449, 25.10.27. Appl., 16.12.26).—Liquid hydrocarbons are caused to flow longitudinally over a heated surface maintained at progressively increasing temperatures, and the moving liquid is caused by convection to undergo a number of local circulations in a vertical and transverse direction with respect to the main oil flow. The evolved vapours are withdrawn.

C. O. HARVEY.

Oil-distilling apparatus. E. C. D'YARMETT, Assr. to O. K. HERNDON (U.S.P. 1,646,448, 25.10.27. Appl., 2.4.26).—Entrained liquid is removed from vapours by passage through an inclined dephlegmator having upwardly and downwardly extending baffles arranged to vary intermittently the velocity of vapour and liquid.

C. O. HARVEY.

Cracking of oils. W. C. KIRKPATRICK, Assr. to NAT. REFINING PROCESS CORP. (U.S.P. 1,646,543, 25.10.27. Appl., 27.8.25).—Hydrocarbons are heated at about 260—316°, and forced at 600—700 lb./sq. in. through a nozzle which discharges into a still maintained at 150—200 lb./sq. in. and about 416°. The still is provided with means for discharging the resulting vapours and carbonaceous deposit.

C. O. HARVEY.

Cracking still [for hydrocarbons]. A. A. PHELAN (U.S.P. 1,646,929, 25.10.27. Appl., 1.11.26).—The top of a vertical cylindrical tank carries an inlet for heated liquid and a vapour outlet, the tank being provided with a partition extending from top to bottom dividing it into a liquid receiving chamber and a vapour outlet chamber intercommunicating through apertures in the lower end of the partition. The former chamber is provided with downwardly-inclined baffles spaced from the walls of the tank, and the latter chamber is partitioned off into a carbon-precipitating compartment (provided with baffles), from the base of which precipitated carbon admixed with liquid is withdrawn, and a liquid overflow compartment provided with an outlet controlled by a liquid-level valve.

C. O. HARVEY.

Apparatus for cracking [hydrocarbon] oil. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,649,105, 15.11.27. Appl., 11.2.21).—Oil, heated in a tube still, passes through a transfer line into an expansion chamber, the discharge end of the transfer line being subdivided into a number of internally-rifled nozzles which cause the oil to emerge into the expansion chamber at a high velocity in a number of circulatory paths.

C. O. HARVEY.

Treatment of hydrocarbon oils. G. EGLOFF and R. T. POLLOCK, Assrs. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,649,104, 15.11.27. Appl., 9.12.20. Renewed 5.2.27).—Relatively heavy asphaltic and emulsified oils (*d* 0.933 or over) are distilled under reduced pressure, heat being applied to the upper surface of the oil by means of an electrically-heated element. C. O. HARVEY.

Treatment of emulsified [hydrocarbon] oils. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,649,102—3, 15.11.27. Appl., [A] 18.9.20, [B] 23.10.20. Renewed, [A] 12.2.27, [B] 15.2.27).—(A) The oil is heated at a temperature adequate for dehydration and then cracked under pressure at a higher temperature. (B) The oil is forced, by means of a reciprocating piston, through a number of spaced foraminated partitions situated within a horizontal cylindrical chamber. C. O. HARVEY.

Refining [centrifugal separation of] lubricating oils. O. C. BREWSTER, Assr. to SHARPLES SPECIALTY CO. (U.S.P. 1,649,095—6, 15.11.27. Appl., [A] 18.10.21, [B] 19.11.21).—(A) Mineral oil is separated centrifugally from waxy constituents, and a carrier liquid at a temperature high enough to melt the wax is fed to the centrifugal rotor at the place of discharge of the latter. (B) A centrifuge for carrying out the process described in (A). C. O. HARVEY.

Retort for treating oil shale. E. B. ROTH (U.S.P. 1,649,195, 15.11.27. Appl., 30.6.26).—A vertical, double-walled retort is divided by floors into several compartments, each compartment being in communication with the gas space between the walls of the retort. A rotating vertical shaft carries agitating arms which extend into the compartments, and each compartment communicates by means of pipes with a chamber for receiving heated gases, situated around and opening into the lowest compartment. C. O. HARVEY.

Recovery of petroleum from oil-bearing sands. H. ATKINSON, Assr. to H. ATKINSON, F. K. HOLMESTEAD, and J. B. ADAMS (U.S.P. 1,651,311, 29.11.27. Appl., 4.4.26).—An alkaline solution, of strength sufficient to displace the oil, is introduced into the sand, which has previously been saturated with water.

H. ROYAL-DAWSON.

Fuller's earth treating process. R. R. ROSENBAUM (U.S.P. 1,649,193, 15.11.27. Appl., 29.1.27).—Fuller's earth is revived by washing it with liquid sulphur dioxide. C. O. HARVEY.

Liquid fuel. H. PLAUSON and P. SCHRÖDER, Assrs. to L. W. BATES (U.S.P. 1,647,471, 1.11.27. Appl., 26.8.21. Austr., 16.2.14).—See B.P. 4340 of 1914; B., 1915, 948.

Automatically-cleaned tar separators. COMP. POUR LA FABR. DES COMPTEURS ET MATÉRIEL D'USINES À GAZ (B.P. 272,237, 7.6.27. Fr., 4.6.26).

Froth flotation apparatus (G.P. 443,616).—See I. **Manufacture of sulphur** (B.P. 256,638).—See VII. **Linings for petrol tanks** (B.P. 279,291).—See XIV.

III.—ORGANIC INTERMEDIATES.

Alcohol from vegetable matter. CARRIÈRE.
Alcohol from sulphite spirit. SCHLUMBERGER.—See V.

PATENTS.

Production of carbon disulphide from its elements. I. G. FARBENIND. A.-G., Asses. of CHEM. FABR. GRIESHEIM-ELEKTRON (B.P. 260,969, 11.10.26. Ger., 3.11.25. Addn. to B.P. 174,040; B., 1922, 708 A).—In the manufacture of carbon disulphide by electrical heating with a movable electrode suspended vertically and a stationary bottom electrode, the latter is constructed as a crucible in which molten slag accumulates, ensuring a more regular passage of current, and forming a heat accumulator as well as preventing the carbon crucible from reacting with sulphur. W. G. CARRY.

Production of concentrated acetic acid. E. VON RETZE, Assr. to HOLZVERKOHLUNGS-IND. A.-G. (U.S.P. 1,647,676, 1.11.27. Appl., 14.6.26. Ger., 17.6.25).—Acetic acid in dilute aqueous solution is esterified, *e.g.*, to the ethyl ester, which is separated, and hydrolysed with a limited amount of water. T. S. WHEELER.

Manufacture of aromatic acid anhydrides. BRIT. DYESTUFFS CORP., LTD., J. B. PAYMAN, and N. HALL (B.P. 280,373, 29.11.26).—A compound of the type of benzotrichloride is heated with 1.5 mols. of water at 100—115° in presence of a suitable catalyst, *e.g.*, zinc chloride, ferric chloride, ferrous sulphate, organic sulphonates or other salts of zinc, copper, iron, aluminium, tin, etc. Crystallised ferrous sulphate may be used to provide both the water and the catalyst. C. HOLLINS.

Manufacture of aliphatic acid anhydrides. II. DREYFUS (B.P. 279,916, 1.6.26).—Acetic anhydride does not combine with dry steam, and hence may be obtained from acetic acid of any dilution by passing the acid vapour over a suitable catalyst and condensing anhydride without condensation of water. This is achieved by using a suitable condenser temperature, or preferably by means of a high-boiling solvent (chlorobenzene, *p*-dichlorobenzene, dibenzyl ether, tetrachloroethane, kerosene, glyceryl acetates, phenetole, anisole, cresols, *p*-tolyl acetate), especially such as are insoluble in water and non-volatile in steam. Other aliphatic anhydrides are similarly prepared. [Stat. ref. to B.P. 257,968, 256,664, and 256,663.] C. HOLLINS.

Manufacture of alkynaphthalenes. I. G. FARBENIND. A.-G., Asses. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (B.P. 260,604, 28.10.26. Ger., 28.10.25).—Naphthalene is alkylated by treatment with an alkyl halide in presence of a metal halide and a metal oxide as acid-binding agent. Even methyl and ethyl chlorides react readily under these conditions at 160—180° (pressure 42—45 atm. with methyl chloride, 26 atm. with ethyl chloride). C. HOLLINS.

Manufacture of 1-diazoanthraquinone-2-carboxylic acids. I. G. FARBENIND. A.-G. (B.P. 262,119, 25.11.26. Ger., 28.11.25).—When the anthraquinone-1:2-isooxazoles of B.P. 147,001 (B., 1922, 50 A) are treated with nitrous acid or substances yielding nitrous

acid (e.g., nitrosylsulphuric acid), they are converted into 1-diazoanthraquinone-2-carboxylic acids. 1-Diazoanthraquinone-2-carboxylic acid is obtained in solution as sulphate or hydrochloride. 5-Nitro-1-diazoanthraquinone-2-carboxylic acid, prepared as crystalline sulphate from the 5-nitro-isooxazole, is converted by cuprous chloride into 1-chloro-5-nitroanthraquinone-2-carboxylic acid, m.p. 301°.

C. HOLLINS.

Manufacture of sodium formate-formic acid compounds. E. ELÖD, Assr. to R. KOEPP & Co. (U.S.P. 1,650,984, 29.11.27. Appl., 24.9.23. Austr., 5.9.22).—See G.P. 424,017; B., 1926, 772.

Manufacture of lactic acid esters. H. W. MATHE-SON and K. G. BLAICKIE, Assrs. to CANADIAN ELECTRO PRODUCTS Co. (U.S.P. 1,650,950, 29.11.27. Appl., 4.9.25).—See B.P. 257,907; B., 1927, 796.

Production of emulsions. W. KRANNICH, H. KRZIKALLA, and C. SCHUSTER, Assrs. to I. G. FARBEIND. A.-G. (U.S.P. 1,652,016, 6.12.27. Appl., 10.3.27. Ger., 10.3.26. Renewed 2.11.27).—See B.P. 267,534; B., 1927, 869.

Absorbent materials (B.P. 280,262).—See I.

IV.—DYESTUFFS.

PATENTS.

Manufacture of a greenish-yellow azo dye. O. Y. IMRAY. From I. G. FARBEIND. A.-G. (B.P. 280,436, 5.5.27).—4-Chloro-2-nitroaniline is diazotised and coupled with acetoacetic *p*-chloroanilide to give a greenish-yellow pigment useful for lakes.

C. HOLLINS.

Manufacture of benzanthrone derivatives containing sulphur. I. G. FARBEIND. A.-G., Asses. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (B.P. 259,608, 9.10.26. Ger., 10.10.25).—3:3'-Bisbenzanthronyl sulphide, m.p. 347° (cf. B.P. 256,059; B., 1926, 867), is obtained by heating 3-bromo- or 3-chloro-benzanthrone with hydrogen sulphide, sodium thiosulphate, sulphur and sodium acetate, or other sulphurising agent (excluding sulphides or polysulphides). C. HOLLINS.

Brown substituted benzidine wool dyes. W. NEELMEIER, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,646,793, 25.10.27. Appl., 16.9.26. Ger., 25.9.25).—Tetrazotised 2:2'-dihalogeno- or -alkyl-benzidines are coupled with a diaminobenzenedisulphonic acid (1 mol.), and with a 7-amino- α -naphthol-3-sulphonic acid, or a derivative in which a hydrogen atom of the amino-group is substituted by alkyl or aryl, to give soluble dyes, which yield on wool and silk from an acid bath reddish-brown shades, particularly fast to fulling.

T. S. WHEELER.

Production of substantive dyes of the stilbene series fast to alkali. CHEMICAL WORKS FORMERLY SANDOZ [CHEM. FABR. VORM. SANDOZ] (B.P. 263,192, 18.12.26. Ger., 19.12.25).—The dyes obtained by condensing aminoazo-compounds with dinitrostilbene- or dinitrodibenzyl-disulphonic acid or with the dyes prepared by action of alkali on *p*-nitrotoluene-*o*-sulphonic acid are oxidised, e.g., with chlorine or hypochlorites, to give direct cotton dyes fast to alkali. Examples are

the oxidation of condensation products from dinitrostilbenedisulphonic acid and metanilic acid \rightarrow cresidine, or sulphanilic acid \rightarrow α -naphthylamine, and from Curcumine S and aminoazotoluenedisulphonic acid.

C. HOLLINS.

Manufacture of triarylmethane dyes. I. G. FARBEIND. A.-G. (B.P. 275,609, 27.7.27. Ger., 9.8.26).—Triarylmethane dyes containing as one of the aryl groups a 4-alkoxydiphenylamine residue show increased light-fastness. Examples are: tetramethyldiaminobenzophenone condensed with *p*-methoxydiphenylamine and sulphonated (blue), and Guinea Green 2G condensed with *p*-phenetidine (blue).

C. HOLLINS.

Manufacture of a greenish-yellow azo dye. H. WAGNER and A. FUNKE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,644,003, 4.10.27. Appl., 8.10.25).—See B.P. 280,436; preceding.

Vat dyes of the anthraquinone series. M. KUGEL, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,651,461, 6.12.27. Appl., 23.3.25. Ger., 31.3.24).—See B.P. 231,532; B., 1925, 910.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

De-suinting and scouring of wool. E. JUSTIN-MUELLER (Bull. Soc. Ind. Mulhouse, 1927, 93, 459–461).—Treatment (de-suinting) of raw wool with water at 55–60° for 5–7 min., and then two successive treatments (scouring) of 5 min. each (with squeezing between) at 45° with an alkaline solution containing 0.2% of Marseilles soap and 0.2% of soda ash resulted in losses of 47.9 and 51.2%, respectively. Microscopical examination indicated that the epithelial scales of the raw wool fibres were invisible owing to a superficial covering of amorphous and crystalline impurities, but immediately after treatment with water they became visible although the crystalline impurities closely resembling ischolesterol remained attached. By de-suinting with warm water and scouring with soap, the resulting losses were 13.0% of fatty substances other than waxes (e.g., lanoline) and 3.1% of wax substances (e.g., cholesterol and ischolesterol) respectively. Raw wool contains sufficient potash so that by treatment with warm water the whole of the fatty substances other than waxes are removed. Soap is necessary in the scouring of wool since aqueous solutions of sodium carbonate are unable to emulsify the waxes present.

A. J. HALL.

Moisture relations of colloidal fibres. J. J. HEDGES (J. Text. Inst., Spec. Issue, 1927, 18, 350–359 T).—The percentage regain of wool obtained by use of a commercial oven is always lower than that given by a laboratory method. A Bradford Conditioning House oven gives results as near the true moisture content as can be obtained without circulating dry air through the oven, the attraction for the last traces of moisture being very great. Curves for the variation of moisture content of chemically-pure wool with atmospheric R.H. are of the usual sigmoid character given by other hygroscopic colloids. The heat of wetting of dry wool is considerable, being 24.1 g.-cal./g. Wool and its absorbed water may be considered as a two-phase system in which a little water is adsorbed by the colloid particles, whilst the bulk is held in a system of pores. The differ-

ence between the apparent (in water) and true specific volumes of wool is much greater than that between the corresponding values for cotton, which, in conjunction with the relative values of the saturation moisture contents, points to a much greater contraction in total volume for wool. Wool, artificial silk, and silk (except heavily weighted silk) are perfect electric insulators when dry and all, except chemically-pure wool, increase in conductivity with increasing humidity. The fastness to light of dyed fabrics also appears to depend on the amount of moisture present. B. P. RIDGE.

Preparation of cross-sections of artificial silk. A. HERZOG (Textilber., 1927, 8, 429—430).—A bundle of artificial silk fibres are coated with thick collodion or a solution of celloidin before embedding in the paraffin wax, a slight twisting of the bundle being also desirable, since it increases the rigidity of the cross-sections. If the sections are not stained, improved definition is obtained by means of oblique illumination under the microscope. A. J. HALL.

Viscose. XII. G. KITA, R. TOMIHISA, K. NAKAHASHI, and J. ONOHARA (J. Cellulose Inst., Tokyo, 1927, 3, 265—273).—In the progressive stages of ripening, the capacity of the viscose to give a continuous thread varies with the temperature of the coagulating bath between 30° and 60° in the sense that the ability to spin at the lower temperatures covers a much smaller range of ripeness than at the higher temperatures. With under-ripe viscose this is due to difficulty of coagulation, whereas with over-ripe viscose, with rapid coagulation the threads become too fine to withstand the tension. A high temperature lowers the viscosity and facilitates the extrusion, but at the same time it increases the rate of coagulation so that the filaments may be finer. The tenacity of threads from the same viscose is generally greater the finer the filament, but the extensibility varies in the contrary direction. The properties of the thread change with the degree of ripening, for which there is an optimum point. In an ammonium sulphate-glucose bath, with sulphuric acid ranging from 4 to 12%, an under-ripe viscose first becomes capable of spinning with the higher concentration of acid, and with the higher concentrations an over-ripe viscose first ceases to give a thread. The threads spun from under-ripe viscose in strongly acid baths are lustreless and voluminous. A similar relation exists with high concentrations of ammonium sulphate as with high concentrations of sulphuric acid, except that the threads spun from under-ripe viscose with strongly saline baths are lustrous; they have also good tenacity. With weaker concentrations of ammonium sulphate the quality of the threads depends more intimately on the attainment of the optimum degree of ripening. J. F. BRIGGS.

[Physical] properties of artificial silk filaments consisting of mixtures of nitrocellulose and cellulose acetate. H. EVERS (Textilber., 1927, 8, 873).—The dry and wet tensile strengths and extensibilities of artificial silk filaments dry-spun from 18% solutions in acetone of mixtures of nitrocellulose (containing 11·33% N) and cellulose acetate were compared. The dry tensile strength of the filaments decreased with an increasing proportion of cellulose acetate, the wet tensile strengths

being about 5% less. The extensibilities of the dry filaments were inappreciably affected by change in composition; when wetted, they swelled only slightly, but had an increased extensibility of 12%. The inflammability of a cellulose acetate filament containing but 5% of nitrocellulose was equal to that consisting entirely of nitrocellulose. A. J. HALL.

Rapid method for determining the degree of digestion of cellulose. C. B. BJÖRKMAN (Papier-Fabr., 1927, 25, 729—730).—A modification of the permanganate method is described, according to which 150 c.c. of 0·02N-permanganate and 5 c.c. of N-sulphuric acid are placed in a beaker at 25° and agitated by means of a glass stirrer operated by a motor at 1000 r.p.m. To this liquid a quantity of squeezed moist pulp containing about 60% of moisture and corresponding to 2 g. of dry cellulose is added while the stirrer is running, and the time of action is accurately gauged by a stop-watch at exactly 30 sec. At that moment 100 c.c. of 0·02N-acid ferrous ammonium sulphate are added and washed in with 40 c.c. of water, the stirrer being then stopped. The total quantity of liquid in the test-beaker being known, an empty Gooch crucible is pressed down into the mass and 100 c.c. of clear liquid are withdrawn through it by a pipette. The excess of ferrous sulphate is determined by titration with permanganate, and the oxygen consumed by the pulp is thence calculated. The results may be converted into the standard chlorine consumption values by reference to a graph, the whole test requiring only about 3 min. J. F. BRIGGS.

Ethyl alcohol from vegetable matter. CARRIÈRE (Bull. Soc. d'Encour., 1927, 126, 612—617).—The problem of the conversion of cellulose into sugar, particularly from vine shoots and twigs, by the action of mineral acids, with the subsequent production of alcohol, is reviewed, reference being made to the Prodor, Meunier, and Dupont de Nemours processes, and to others in operation in Sweden and Germany. In the saccharification of vine stems etc. the results are dependent on time, and on the concentration and nature of the acid used, the best results being obtained by using a 1% solution of hydrochloric acid; after 7 hrs. the sugar obtained is 35·4% on the dry raw material. With sulphuric acid of similar concentration, even after 19 hrs., the sugar yield is only 32%. The quantity of sugar produced is 50% more from hard wood than from soft. The broken-up raw material is boiled for 4 hrs. with seven times its weight of 1·2% hydrochloric acid, the saccharine liquor separated from the residue, and the liquid boiled for a further 4 hrs. in contact with fresh raw material; this process is repeated in all four times, after which the solution is separated from the residue. The solution after filtration is neutralised and fermented. The amount of sugar in the solution is determined by means of Fehling's solution. H. M. LANGTON.

Explosion risks in the industrial preparation of absolute alcohol from sulphite spirit. E. SCHLUMBERGER (Papier-Fabr., 1927, 25, 704).—The explosion range of alcohol-air mixtures is considerably greater than that of benzene-air mixtures, and although the heat of combustion of alcohol is less than half that of benzene vapour, the heats of combustion of explosive

mixtures of the two substances with air are nearly equal (about 475 kg.-cal./litre), as are also the theoretically possible explosion temperatures (about 1340°) and the ignition temperatures (about 600°). Precautions such as the use of an inert gas should therefore be taken in the manufacture of absolute alcohol from sulphite spirit, especially when the alcohol retained by the lime used for dehydration is removed in an air-stream.

W. J. POWELL.

De-inking of paper. T. R. BRIGGS and F. H. RHODES (Fourth Colloid Symposium Monograph, 1926, 311—327).—The "apparent" degree of de-inking of newspaper was determined as follows (30—40 min., 50 g. in 1 litre with paddle 1300 r.p.m.): sodium oleate (5 g.), 100; fuller's earth (10 g.) in 0.02*N*-sodium hydroxide, 95; 0.1*N*-sodium silicate, 84; sodium resinate (5 g.), 83; 0.1*N*-sodium hydroxide, 82; 0.02*N*-sodium hydroxide, 78; 0.1*N*-borax, 75; gelatin in 0.02*N*-sodium hydroxide, 71; sodium hydroxide (70—55°), 70; 0.1*N*-sodium carbonate, 69; gum arabic (10 g.) 65; gelatin (10 g.), 55; 0.1*N*-sucrose, 46; distilled water, 30; egg-albumin (10 g.), 26; 0.1*N*-sodium, calcium, or aluminium chloride, or lime water, 0. The ideal de-inking agent is a substance, preferably colloidal, which is adsorbed much more strongly by ink than by paper; if the reverse is the case, a mortanting action results. CHEMICAL ABSTRACTS.

Pulping of pine wood by the sulphite process. E. HÄGGLUND (Cellulosechem., 1927, 8, 111—112).—A reply to the criticisms of Schwalbe and Berndt (cf. B., 1927, 519).

W. J. POWELL.

Cellulose esters. G. KITA, I. SAKURADA, and T. NAKASHIMA (Cellulosechem., 1927, 8, 105—110, and Sci. Papers Inst. Phys. Chem. Res. Tokyo, 1927, 6, 197—213).—See B., 1926, 944.

Hydrolysis of starch. FARGHER and PROBERT.—See XVII.

PATENTS.

Manufacture of felt. H. HAAKH (E.P. 275,939, 7.6.27. Ger., 14.8.26).—The manufacture of, e.g., hats from felt is facilitated by treating the felt at any desired stage with chlorinating or brominating agents; the liquor resulting from the electrolysis of a solution of sodium chloride containing 5 g. of chlorine per kg. of felt can thus be used.

D. J. NORMAN.

Treatment of fibrous materials. T. L. DUNBAR, Assr. to STEBBINS ENGINEERING & MANUF. Co. (U.S.P. 1,646,084, 18.10.27. Appl., 3.9.26).—In a pulping process, fresh liquor heated to a predetermined temperature is pumped into the digester during the cooking process in order to build up a relatively high pressure while maintaining a relatively low temperature in the digester.

D. J. NORMAN.

Treatment of fibrous vegetable material. R. FORSYTH, Assr. to H. J. RODGERS (U.S.P. 1,646,087, 18.10.27. Appl., 7.2.24).—The material is treated with hot distilled water containing a very small quantity of hydrochloric acid, squeezed to remove gums etc., and treated with hot distilled water containing a small percentage of sodium carbonate. After further squeezing, the material is rinsed with boiling distilled water, dried

slowly at a moderate temperature, and the bast fibres are mechanically separated from woody matter.

D. J. NORMAN.

Manufacture of fibrous compounds and articles moulded therefrom. JAROSLAW'S ERSTE GLIMMER-WAREN-FABR., and R. SCHROEDER (E.P. 278,038, 26.5.26).—A fibrous material capable of being moulded into non-hygroscopic products of high mechanical strength and in which each individual fibre is coated with a binding medium, is prepared by coating or impregnating paper or fabric with a synthetic resin and afterwards completely disintegrating it by scutching it in high-speed mills etc. so that the fibres are separated but not broken; further binding agents may be added to the disintegrated material.

A. J. HALL.

Manufacture of artificial silk. E. ELSÄESSER and A. HARTMANN, Assrs. to AMER. BEMBERG CORP. (U.S.P. 1,646,788, 25.10.27. Appl., 8.4.24. Ger., 7.12.23).—The threads are spun into the precipitating liquid in the inner of two inverted co-axial cones, and the liquid is fed to the upper portion of the outer cone. Formation of currents, which tend to break the silk as spun, is avoided.

T. S. WHEELER.

Spinnerette [for artificial silk]. R. V. WILLIAMS (U.S.P. 1,647,822, 1.11.27. Appl., 12.10.25).—The spinnerette is composed of an alloy containing 80—99% Au and 20—1% Ni.

T. S. WHEELER.

Production of non-glowing paper. M. LANGE and L. KAISER (G.P. 443,766, 28.4.25).—The paper is impregnated with salts of the rare-earth metals, e.g., with a mixture of thorium and cerium salts.

L. A. COLES.

Manufacture of cellulose esters. P. BERTHON, Assr. to SOC. DE STEARINERIE & SAVONNERIE DE LYON. (U.S.P. 1,651,990—1, 6.12.27. Appl., [A] 12.2.24, [B] 8.5.25. Fr., [A] 31.7.23).—See B.P. 219,926; B., 1925, 37.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing [of cotton] with Indanthrene Blue GCD. G. DURST and H. ROTH (Textilber., 1925, 6, 837—839).—Small-scale experiments supplemented by large-scale trials in jigs without submerged rollers show that in dyeing cotton with Indanthrene Blue GCD at 60°, the dye is uniformly absorbed during the first 30 min., and that subsequent absorption up to 1 hr. is much slower and small. During the first 5 min. a decrease (about 20%) of alkalinity of the dye liquor occurs due to adsorption by the cotton, but no further loss occurs during the remainder of the dyeing process. The unavoidable absorption of atmospheric oxygen accompanied by a corresponding loss of hyposulphite which occurs during dyeing is greater the higher the temperature of the dye liquor; under comparative conditions, a cold dye liquor lost 1% of its hyposulphite per hour during a period of 26 hrs., but at 60° the corresponding loss was 18.5% per hour during 3 hrs. The absorption of Indanthrene Blue GCD by cotton is most rapid at 45°; increase of temperature of the dye liquor above 60° produces an inappreciable increase in the rate of absorption.

A. J. HALL.

[Dyeing of cotton with] Indanthrene Blue RS. G. DURST and H. ROTH (Textilber., 1927, 8, 158—160).—The methods previously described (cf. B., 1926, 819) for determining the composition of Indanthrene dye vats have been used for following the changes which occur in dye vats containing Indanthrene Blue RS during the dyeing of cotton. The rate of absorption of dye by the cotton is most rapid during the first 15 min., Indanthrene Blue RS thereby behaving differently from Indanthrene Blue GCD as determined by earlier investigations (see preceding abstract). Similarly, a marked loss of hyposulphite and caustic soda occurs during the first 5 min. of dyeing. The methods of analysis have proved sufficiently accurate and rapid to allow their application to the control of the composition of Indanthrene dye vat liquors used for padding cotton fabrics.

A. J. HALL.

[Dyeing with] Indanthrene dyes. G. DURST and H. ROTH (Textilber., 1927, 8, 785—787).—Curves are given showing the changes in the concentrations of dye and sodium hyposulphite which occur in dye liquors containing Indanthrene Yellow GK, Indanthrene Brown R, or Indanthrene Orange RRTS, when used for dyeing cotton under various conditions of time and temperature, the methods of analysis used being those previously described (B., 1926, 819). The rate of absorption by cotton of a "cold-dyeing" dye (e.g., Indanthrene Orange RRTS) is much less affected than that of a "hot-dyeing" dye (e.g., Indanthrene Brown R) by a change in the temperature of the dye liquor. The minimum loss of hyposulphite produced by atmospheric oxidation occurs in dye liquors containing about 5 g./litre of sodium hyposulphite.

A. J. HALL.

[Printing of] green discharges on [indigo-]blue ground. R. HALLER (Sealed Note 1861, 18.9.08. Bull. Soc. Ind. Mulhouse, 1927, 93, 456—457). Report by C. VAUCHER (*Ibid.*, 457—458).—Excellent green discharges are obtained by printing cotton fabric dyed with indigo with a discharge paste consisting of 540 g. of a 50% solution of gum, 150 g. of Rongalite, 86 g. of aniline, 110 g. of tannic acid, 70 c.c. of water, 16 g. of Acridine Yellow, 10 g. of Methylene Blue B, and 124 g. of 96% alcohol, then steaming for 5 min. in a Mather-Platt in the absence of air (oxygen), passing for 1 min. through a vat containing a boiling liquor consisting of 1000 litres of water, 25 litres of a 10% solution of soap, and 6 litres of an alkaline solution of glycerin and tartar emetic (3 litres of water, 2 kg. of glycerin, 500 g. of tartar emetic, and 1 litre of caustic soda, *d* 1.16), the rollers of the vat being submerged so that reoxidation of the indigo-white in the discharged parts is avoided. Subsequently the fabric is washed thoroughly so that any reduced Methylene Blue is oxidised, and then soaped and washed. Pure white discharges may be obtained simultaneously by use of a discharge paste consisting of 1 litre of a 50% solution of British gum, and 150 g. of Rongalite. Vaucher reports favourably on the process.

A. J. HALL.

Printing of Alizarin Red on non-prepared fabric. LA MANUF. SCHEIBLER [with A. LISZKOWSKI] (Sealed Note 1886, 1.3.09. Bull. Soc. Ind. Mulhouse, 1927, 93, 449—450). Report by P. WILHELM (*Ibid.*, 451).—

In printing Alizarin Red the usual preparation of the cotton fabric with Turkey-red oil is dispensed with and 10—15% of a compound of ricinoleic acid and aniline is added to the printing paste. *E.g.*, a very bright red shade is obtained by printing fabric with a paste consisting of 45.3 pts. of a starch thickening (prepared with 26 pts. of wheat starch, 5 pts. of acetic acid of *d* 1.036, and 51 pts. of water), 3 pts. of Alizarin 1D (B.A.S.F.) 20% paste, 12 pts. of Alizarin GFX (Bayer) 20% paste, 10 pts. of aluminium thiocyanate (*d* 1.116), 3 pts. of aluminium nitrate (*d* 1.116), 8 pts. of calcium acetate (*d* 1.116), 1.2 pts. of stannic oxalate (*d* 1.125), 2 pts. of tartaric acid 15%, 3.5 pts. of 60% formic acid, and 12 pts. of the product obtained by heating 300 pts. of ricinoleic acid with 95 pts. of aniline (this product is soluble in ammonia). Wilhelm reports that the resulting shades are as bright and fast to soaping as those obtained by the usual method on oil-prepared fabric. During steaming, the aniline neutralises the acids present and thus prevents tendering of the fabric.

A. J. HALL.

New reserve effects under Aniline Black [by printing]. G. KNOOP (Sealed Note 1889, 16.3.05. Bull. Soc. Ind. Mulhouse, 1927, 93, 452—453). Report by P. BINDER (*Ibid.*, 453—455).—Reserve effects under Aniline Black, as clearly defined as those produced by the usual Prud'homme method, are obtained by printing cotton fabric with a reserve paste containing a gum thickening, zinc oxide, and Turkey-red oil, drying, then padding with the usual "prussiate" Aniline Black liquor, ageing in a small Mather-Platt, and after-chroming. Coloured effects are obtained by adding basic dyes (Thioflavine T, Brilliant Green, Victoria Blue BO, and Turquoise Blue are suitable) to the reserve paste. A satisfactory reserve consists of 1 kg. of gum thickening, 370 g. of zinc oxide, 500 g. of Turkey-red oil, 30 g. of ammonia solution (*d* 0.915), and 50 g. of a basic dye. Binder reports favourably on the process especially as regards the production of clear-cut edges to the pattern effects.

A. J. HALL.

PATENTS.

Dyeing [regenerated cellulose materials]. BRIT. DYESTUFFS CORP., LTD., and J. BADDILEY (B.P. 280,320, 7.9.26).—Level shades on viscose silk are obtained by using monoazo dyes containing as coupling component 2-amino-5-naphthol-7-sulphonic acid ("J-acid") or its *N*-substituted derivatives. Eighteen examples are mentioned, in which J-acid, its *N*-phenyl, *p*-tolyl, *o*-anisyl, β -naphthyl, and benzoyl derivatives, and 5-hydroxy-2-naphthylglycine-7-sulphonic acid are coupled with diazotised aniline, *p*-chloroaniline, *p*-nitroaniline, *p*-aminoacetanilide (with subsequent hydrolysis), *o*-anisidine, 2-chloro-*p*-toluidine-5-sulphonic acid, dehydrothio-*p*-toluidinesulphonic acid, β -naphthylamine, β -naphthylamine-6-sulphonic acid and -6:8-disulphonic acids. The shades are orange to red and violet. C. HOLLINS.

Colouring of furs and other materials. BRIT. DYESTUFFS CORP., LTD., R. S. HORSFALL, and L. G. LAURIE (B.P. 279,942, 4.8.26).—The solubility of dyes of all classes in aqueous liquids is greatly enhanced by the presence of hydroxylated benzene derivatives, *e.g.*, phenol, pyrogallol, and particularly resorcinol. The

deeply-coloured solutions are used for colouring furs, bone, ivory, horn, resins, and celluloid films.

C. HOLLINS.

Colour printing on fabrics [containing cellulose acetate]. ETABL. PETITDIDIER (ANC. MAISON JOLLY-BELIN) (B.P. 256,238, 27.7.26. Fr., 1.8.25).—Fabrics containing cotton (or viscose silk) and acetate silk are printed with a colour having affinity for acetate silk, steamed, and rinsed, and the whites finally cleared with 0.1% solution of sodium hyposulphite. For the printing pastes, Duranol or Setacyl colours are diluted with water and thickened with gum etc.; basic colours are used with "acetanol" (a pyridine salt manufactured by Etabl. Kuhlmann); Ionamines are excluded. C. HOLLINS.

Colour printing on fabrics having a foundation of silk or wool. ETABL. PETITDIDIER (ANC. MAISON JOLLY-BELIN) (B.P. 258,562, 6.8.26. Fr., 17.9.25. Addn. to B.P. 256,238; preceding).—In the application of the prior invention to fabrics of acetate silk and silk the whites are cleared by means of an acid bath (0.1% sulphuric acid) or by means of 0.2–0.3% sodium hyposulphite solution. For acetate silk-wool mixtures the hyposulphite solution may be 0.5%. C. HOLLINS.

Manufacture of wetting-out agents etc. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, and E. CHAPMAN (B.P. 279,990, 20.9.26).—Wetting-out agents are obtained by sulphonation with 100% sulphuric acid or weak oleum of a mineral oil fraction, b.p. 240–300°. C. HOLLINS.

Mordant paste. S. NIISATO (U.S.P. 1,647,539, 1.11.27. Appl., 21.4.27).—A mixture of a sodium soap, tannic acid, acetic acid, sodium chloride, and the solution obtained by extracting rice bran with boiling water is claimed. T. S. WHEELER.

Discharge effects on [textile] materials containing acetyl cellulose. L. SMITH, ASSR. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,651,639, 6.12.27. Appl., 23.9.26. U.K., 25.11.25).—See B.P. 262,254; B., 1927, 71.

Preparation of cotton fibres [for direct dyeing]. P. KARRER, ASSR. to CHEMICAL WORKS FORMERLY SANDOZ (U.S.P. 1,650,989, 29.11.27. Appl., 6.3.26. Ger., 11.3.25).—See B.P. 249,842; B., 1927, 71.

Treatment of articles with soaps (B.P. 253,105 and 280,110).—See XII.

Decoration of fabrics (G.P. 441,690).—See XXI.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Volumetric and thermal relations of contact sulphuric acid. R. NITZSCHMANN (Continental Met. Chem. Eng., 1927, 2, 176–178, 208–209).—Equations are given for the calculation of the volumes of oxygen, nitrogen, and sulphur dioxide as a function of the percentage conversion and the percentage of sulphur dioxide in the initial burner gas. Data are also given for the heat content of roaster gases (from iron pyrites) as a function of the theoretical temperature of combustion, the maximum temperature difference between the gases entering and leaving the converter as a function of the percentage conversion, the temperature of the

gases leaving the contact catalyst as a function of the percentage conversion, and the relations of the heat exchange up to emergence from the catalyst.

CHEMICAL ABSTRACTS.

Determining heat consumption for caustic [soda and potash] dehydration. G. ANGEL (Chem. Met. Eng., 1927, 34, 683–685).—Whilst the heat absorbed in the separation of solid caustic soda or potash from solution is equal to the heat liberated on dissolution, and the heat for evaporation of the water is also calculable, the question of heat absorbed in raising the temperature of the solution to its final b.p. presents difficulties. The problem can, however, be regarded as the determination of the total of the following:—Heat required to raise pure sodium hydroxide to the final temperature, total heat and superheat of saturated steam at this temperature, and the heat of dilution. It is shown that this total can be approximately calculated, using mean values for the specific heats of solid sodium and potassium hydroxides; the results indicate that the efficiency of certain coal-fired caustic pots was 46½%. Electrically-heated pots may have 85% efficiency. Dehydration *in vacuo* was shown to effect a saving of only 4% of the heat consumed in the latter case, but with coal-fired pots, owing to indirect savings, the economy is 20%.

C. IRWIN.

Function of steam in the lime kiln. E. E. BERGER (U.S. Bur. Mines, Tech. Paper No. 415, 1927, 43 pp.).—It is usual in lime-burning to place a steam-jet under the grate. This has been generally considered to accelerate the process by specific chemical action, a view based mainly on experiments by Herzfeld. To investigate the point an apparatus was designed by which granulated limestone could be ignited in a quartz tube with constant air supply. The rate of calcination at varying temperatures was then determined with similar feeds of air, steam, and helium, the latter being chosen on account of its absolute inertness. It was found with all three that decomposition commenced at 600°. It was slightly more rapid with steam than with air and still more so with helium. It is concluded, therefore, that the differences are due to varying rates of heat-transfer, and that any chemical action of the steam is excluded. The differences are in accordance with the heat capacity and conductivity of the gases. In the lime kiln where only 5% of steam is used, the effect of these differences must be negligible. Steam, however, by its reactions with carbon cools the fuel bed, it being certain that under the conditions obtaining in practice a large part of it must be reduced in the upper part of the bed. This effect also minimises clinkering of the ash. The combustion of the resulting hydrogen and carbon monoxide with the secondary air give the desirable long flame. It is recommended that only enough steam should be introduced to prevent clinkering. If this is not sufficient to produce the necessary draught a fan should be used rather than excess steam. In some plants waste flue gas is used in place of steam, the effect of carbon dioxide on the fuel bed being very similar to that of steam. It is shown that with 5% of carbon dioxide in the feed gases the thermal loss at the stack is less than with 0.5 lb. of steam per lb. of fuel. The loss rises rapidly if more carbon dioxide is used. C. IRWIN.

Detection of iodides in alkali bromides. S. V. MIKÓ (Pharm. Zentr., 1927, 68, 763—765).—The presence of 0.4 mg. of potassium iodide can be detected in 5 c.c. of a 5% alkali bromide solution by oxidation with 5 drops of 0.1*N*-potassium permanganate in 5*N*-hydrochloric acid solution, and shaking the mixture with 2 c.c. of xylene; the bromine is decolorised within 1 min. and the rose colour of the iodine remains. S. I. LEVY.

Determination of the purity of elementary boron. J. W. ANDREWS (Chemist-Analyst, 1926, No. 47, 16).—The boron is fused in a covered silver crucible with a mixture of sodium hydroxide and potassium nitrate, the product is slightly acidified with hydrochloric acid, and the carbon dioxide boiled off under reduced pressure. The liquid is neutralised (using *p*-nitrophenol as indicator), mannitol is added, and the boric acid is titrated in the usual way, employing phenolphthalein. CHEMICAL ABSTRACTS.

Shale from potash mines. DOMEC.—See II.

Germicidal efficiency of sodium salts. LEVINE and others.—See XXIII.

PATENTS.

Production of nitric acid. METALLBANK U. METALLURGISCHE GES. A.-G., and DEUTS. SPRENGSTOFF A.-G., Assees. of W. MELZER and W. VON BOLTENSTERN (G.P. 443,338, 15.1.26. Addn. to G.P. 440,657; B., 1927, 813).—The oxidation and condensation stages in the process described previously are effected by the action of ozone formed at the electrodes. The quantity of ozone formed can be regulated by varying the electrical potential, and can be augmented from other sources.

L. A. COLES.

Production of anhydrous sodium sulphate from Glauber's salt. FELLNER & ZIEGLER A.-G., and B. YOUNG (G.P. 444,048, 29.8.23).—The salt is dried in a rotating drum of which the first section is conical in shape and is provided with a perforated screw conveyor and with outlets for the expelled water. L. A. COLES.

Manufacture of readily soluble salts of hydrofluosilicic acid. W. MÖLLER and W. KRETH (B.P. 263,780, 7.12.26. Ger., 23.12.25).—A highly concentrated solution of hydrofluosilicic acid is mixed with a quantity of a readily-soluble salt (*e.g.*, magnesium chloride) several times greater than that theoretically required to form the silicofluoride, and preferably in a solid state, the silicofluoride being precipitated in crystalline form. After separation of the mother-liquor, the excess of salts and the acid liberated may be recovered therefrom.

W. G. CAREY.

Conversion of barium carbonate into barium oxide. P. ASKENASY and R. ROSE (G.P. 443,237, 23.1.25).—Barium carbonate is heated with a large excess of carbon, preferably petroleum coke, and during or after the process excess carbon is removed by heating the material in a current of air or sulphur dioxide at a temperature sufficiently high to prevent conversion of the oxide back into the carbonate. L. A. COLES.

Utilisation of barium and strontium sulphates. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G., Assees. of F. ROTHE and H. BRENEK (G.P. 443,320, 13.11.24).—Mixtures of the sulphates with silica or siliceous material

in such quantities that silicates of the type M_2SiO_4 to M_3SiO_5 are formed are heated at the decomposition temperature (1100°) or higher, in an oxidising atmosphere and in the presence of steam, which may be added as such or formed during the process by passing in gases containing hydrogen. Silica recovered on decomposing the products with mineral acids, or residual alkaline-earth metasilicates obtained by decomposing them with water, are used for the decomposition of fresh material. L. A. COLES.

Impregnation of calcium carbide [to protect it against moisture]. OFFICE CENTRAL DE L'ACÉTYLÈNE ET DE LA SOUDURE AUTOGENE. (F.P. 619,959, 5.8.26).—Lubricating oil, after use in internal-combustion engines, is used for the purpose. L. A. COLES.

Production of a basic calcium aluminium nitrate. I. G. FARBENIND. A.-G., Assees. of K. BLUMRICH, P. OSSWALD, and W. GEISLER (G.P. 443,568, 4.12.25).—Calcium oxide is added to calcium nitrate solution containing alumina or aluminium nitrate.

L. A. COLES.

Production of crystalline basic aluminium nitrate. I. G. FARBENIND. A.-G., Assees. of H. FINKELSTEIN (G.P. 444,517, 25.1.25).—A solution of aluminium hydroxide in nitric acid in such proportions that the constitution of the solute lies between the mono- and di-hydroxynitrate, or the acid content of which does not greatly exceed that required by the former, is allowed to crystallise to such an extent that the crystals approximate to the dihydroxynitrate, and, after removal of the crystals, the mother-liquid is used for the dissolution of more aluminium hydroxide. L. A. COLES.

Decomposition of potassium magnesium bicarbonate. VER.FÜR CHEM. U. MET. PRODUKTION (G.P. 443,456, 10.4.25).—The double salt ($KHCO_3 \cdot MgCO_3 \cdot 4H_2O$) is decomposed by treatment with magnesium hydroxide, and, immediately the reaction is complete, carbon dioxide is led into the potassium carbonate solution formed while it is still in contact with the precipitated magnesium carbonate ($MgCO_3 \cdot 3H_2O$). L. A. COLES.

Production of chromium salts free from iron [from mixtures of chromic and ferric salts]. I. G. FARBENIND. A.-G., Assees. of C. MÜLLER and L. SCHLECHT (G.P. 443,624, 24.7.24. Addn. to G.P. 410,927; B., 1927, 546).—Iron is precipitated as ferric hydroxide or as a basic salt, which is removed from the solution, by heating aqueous solutions of the mixed salts at high temperatures, preferably above their b.p.

L. A. COLES.

Production of acid pyrophosphates. J. A. BENCKISER CHEM. FABR., and K. KLINGBIEL (G.P. 443,196, 6.1.22. Addn. to G.P. 410,098; B., 1925, 401).—Primary alkali or alkaline-earth phosphates are heated together with small quantities of oxidising salts of non-volatile bases. L. A. COLES.

Production of arsenates and pyroarsenates. E. BRITZKE (G.P. 443,286, 26.7.25).—A mixture of arsenic trioxide vapour with an excess of air or oxygen is passed over bases at 500° or 800° for the production of arsenates or pyroarsenates, respectively. L. A. COLES.

Recovery of carbon dioxide from gas mixtures. EISEN- U. STAHLWERK HOESCH A.-G. (G.P. 443,684,

17.10.25).—The gases are passed through alkali solutions, and carbon dioxide is subsequently liberated from solution by electrolytic means. L. A. COLES.

Production of solutions and compounds of chlorine. G. ORNSTEIN (G.P. 443,683, 31.7.26).—Liquid chlorine and a solvent or reacting liquid are sprayed, in such a manner that intimate admixture of the particles is effected, into a chamber insulated to prevent loss of heat by radiation. L. A. COLES.

Production of hydrogen bromide from its elements. J. D. RIEDEL A.-G. (G.P. 443,195, 27.4.24).—Bromine vapour and hydrogen are passed together or separately through a heated, perforated or porous device constructed of refractory material resistant to the action of bromine. L. A. COLES.

Manufacture of sulphur. C. STILL, Assee. of GES. F. KOHLENTCHNIK M.B.H. (B.P. 256,638, 6.8.26. Ger., 6.8.25).—Crude material containing sulphur, *e.g.*, coal-gas purification material, is treated with ammonium sulphite solution containing free ammonia in a steam-heated vessel fitted with stirring gear, and, after settlement of the insoluble material, which is again used for gas purification, the ammonium thio-sulphate is run off and kept at 90° for about 8 hrs. with a measured amount of sulphuric acid, the precipitated sulphur being separated and purified. To obtain the ammonium sulphite, the crude material, with the addition of pyrites, is roasted in a rotary furnace and the resulting sulphur dioxide is purified, cooled, and absorbed in ammonia in a vertical scrubber. W. G. CAREY.

Production of precipitated material containing silicon [for coating articles]. SIEMENS & HALSKE A.-G., Assees. of E. KRAUSE (G.P. 444,363, 21.10.25).—A mixture of a non-reacting gas, *e.g.*, carbon monoxide, with a hydrocarbon derivation containing silicon, *e.g.*, silicon tetraethyl, is passed over the heated article upon which it is desired to form a siliceous deposit. L. A. COLES.

Production of arsenic acid and its solutions. P. ASKENASY and E. ELÖD (U.S.P. 1,650,860, 29.11.27. Appl., 29.1.25. Ger., 29.3.24).—See B.P. 255,522; B., 1926, 823.

VIII.—GLASS; CERAMICS.

Frosting of glass by mixtures containing hydrofluoric acid and alkali fluorides. C. D. SPENCER and L. ORT (J. Amer. Ceram. Soc., 1927, 10, 403—410).—The crystal-like areas on acid-frosted glass, which are detected by microscopic examination at high magnifications, have been explained by three theories. The first assumes that the glass has a crypto-crystalline structure, the second that etching results from the deposition of a crystalline or amorphous precipitate, whilst the third, which is accepted by the authors, postulates that these areas are merely amorphous glass, the appearance of crystallinity being due to the protective action of crystals of silicofluorides during etching, with the consequent selective attack by the solvent. Observations show, in the initial stages of etching, the formation of a finely-divided precipitate at the interface between the glass and the solution. After a short time crystals of silicofluorides make their appearance in the solution

contiguous to the glass surface, and then settle in contact with the glass itself. These crystals expand laterally, and finally establish contact with their neighbours, protecting the glass immediately beneath them from further attack. Thus the glass surface obtains a crystalline appearance by reason of this peculiarity of the acid attack. Surface tension also influences the development of this surface structure. A theory of the mechanical weakness of frosted hollow-ware, based on the physical properties of fissures of small radius of curvature, is put forward. It is suggested that by increasing this radius—by the fortifying action of different solvents—increased strength can be obtained. A. T. GREEN.

Third progress report on investigation of saggar clays; their elasticity and transverse strength at several temperatures. R. A. HEINDL and W. L. PENDERGAST (J. Amer. Ceram. Soc., 1927, 10, 524—534; cf. B., 1926, 539, 917).—The apparatus used in the determination of the elasticity and transverse breaking strength, at three temperatures ranging up to 950°, together with its calibration and precision, is described. Specimen bars, 1 sq. in. in cross-section and about 11 in. long, made of 50% of clay and 50% of grog, were prepared and fired at 1230°, and finally subjected to the tests. The load-deflection curves show that, in the majority of cases, Hooke's law holds good, and that the yield point and point of rupture of clays are, generally, reached simultaneously. One material, however, of high porosity and low strength, shows a definite yield point under a load of 37½ lb., and a point of rupture at 60 lb. The seventeen clays tested can be divided into three groups, showing for the modulus of elasticity (1) a decrease at the highest temperatures; (2) a great increase at the highest temperatures; (3) an increase approximately proportional to the temperature increase. The majority of clays increase in modulus of elasticity when tested at 750° as against room temperatures. Only those clays which were comparatively weak increase greatly in strength at 750°, the majority showing little variation from their corresponding values when cold. An approximately direct relationship exists between transverse strength and elasticity, and an inverse one between porosity and elasticity. With two clays plastic deformation started at 950° with a load of 60 lb./in.² Irrespective of applied load, this was the lowest temperature for plastic deformation. The resistance of a saggar to failure is directly proportional to its modulus of rupture and inversely to its modulus of elasticity and coefficient of linear thermal expansion. This empirical relationship is satisfactory for values obtained between 500° and 700°. A. T. GREEN.

Changes in the viscosity of clay slips and glaze suspensions on ageing and by treatment with electrolytes. J. R. BOWMAN (J. Amer. Ceram. Soc., 1927, 10, 508—516).—The thinning of a glaze suspension or clay slip (*i.e.*, reduction in viscosity on being kept) was examined in the case of two glazes, both showing a strong tendency to thin out—one fritted and the other raw—and of an English ball clay. These slips and glazes were mixed with water and ground in a ball mill. Viscosity was measured by the "pipette" method, precautions being taken to control the temperature. The

viscosity of a thin clay slip decreased slightly during the ageing period, but with a "thick" slip a much greater reduction took place, the minimum being reached after six days. With glaze suspensions a very pronounced decrease in viscosity was noted after two days, the rate of decrease becoming very slow after six days. This spontaneous thinning was greatly accelerated by heat. Glaze suspensions, aged until thinning was complete and then brought back to their original condition of viscosity by means of an electrolyte, were not nearly so liable to a repetition of the phenomenon of thinning, and when thinning occurred it did so to a much less extent. Further viscosity reduction in the acid-treated sample was smaller and less rapid than in the base-treated suspension. The behaviour of glaze suspensions in practice can be controlled either by heating until thinning is completed and then bringing them up "to gauge" with an electrolyte, or by keeping the glaze for several days and then treating it with an electrolyte. The merits of the second method have been proved. A. T. GREEN.

Hydrogen-ion concentration and electrical conductivity of clay slips. I. Plant study. A. H. FESSLER and H. M. KRANER (J. Amer. Ceram. Soc., 1927, 10, 503—507).—To elucidate a method of clay slip control in industrial operations, daily samples of tap water, casting and plastic slips, consignments of clay, and the clay slips in blungers were examined. Measurements of the slip consistency, viscosity, p_H value, and electrical conductivity of many individual samples are reported in graphical form. Although considerable variations in the p_H value of tap water and of casting and plastic slips are noted, these do not correspond with any apparent variation in the workability of the body made from these slips. There is no relation between the p_H value of the tap water used in blunging and the viscosity of the slips. Although different consignments of clay showed differing p_H values, no corresponding differences in the properties of the clay could be detected. Changes in the viscosity and density of plant slips do not necessarily produce changes in their conductivity. Further, hydrogen-ion concentration is not influenced as much as is viscosity by the electrolyte present. Viscosity determination remains the best method of slip control. Methods for determining the p_H value and electrical conductivity are described. A. T. GREEN.

Effect of fine grinding on an indurated clay. T. C. WALKER (J. Amer. Ceram. Soc., 1927, 10, 449—450).—A clay mined in an indurated form was found to lack plasticity in normal use. With the object of testing improvement in this property, the clay was ground to pass a 10, 30, 100, and 150 mesh, respectively. It was found that fine grinding gives a direct increase in the plastic properties, and increases the water of plasticity, tensile strength, and drying shrinkage of the raw clay. The fired samples showed increased shrinkage and a decrease in percentage absorption and porosity, with augmented fineness of the raw clay. A. T. GREEN.

Mechanical introduction of barium into clay bodies. M. W. BLAIR (J. Amer. Ceram. Soc., 1927, 10, 464—466).—Barium carbonate is added to clay bodies to prevent scumming. The amount of this addition, which should always be experimentally determined,

varies from 2 to 20 lb. per thousand bricks. Its addition as a dilute solution forming the pugging water is not efficient since the amount of water required varies with the clay condition, and results in a variable amount of the barium salt being introduced. When added as a concentrated solution, disadvantages arise by reason of the amount of scrap returned to the pug mill and the tendency of barium carbonate to settle out when the solution flows appreciable distances. The sprinkling of the correct amount of the salt on to a raw clay before it is subjected to grinding is inadequate owing to the indefinite nature of the mixing. More accurate methods are based on the principle of feeding the salt into the dry pan in proportion to the speed of the feeding conveyor.

A. T. GREEN.

Bureau of Standards investigation of feldspars—second progress report. R. F. GELLER (J. Amer. Ceram. Soc., 1927, 10, 411—434).—Screen analyses and air elutriation, chemical analyses, softening range, softening point, and true specific gravity determinations on nineteen feldspars are reported. Whiteware bodies, in which these feldspars were the only variable constituents, were made and tested for mechanical strength, colour, light transmission, firing behaviour, and thermal expansion. Glazed specimens were subjected to tests for crazing. Chemical analyses indicate that the feldspars can be divided into three fairly distinct groups. The refractoriness increases and the softening range decreases with increasing K_2O content. The true specific gravity of crystalline feldspar ranges from 2.635 for commercially pure soda feldspar to 2.572 for commercially pure potash feldspar. The volume shrinkage of vitreous bodies appears to decrease with increase in the K_2O content. Whereas the colour of a body in reflected light is not influenced by the feldspar, the translucency increases with increase of the potash content. The mechanical strength of a body is affected more by the method of firing than by the nature of the feldspar. The thermal expansion is increased by rapid firing and by an increased content of soda in the spar. A. T. GREEN.

Solubility of fired [ceramic] bodies in hot sulphuric acid. A. V. BLEININGER (J. Amer. Ceram. Soc., 1927, 10, 535—537).—The assumption that the solubility of an earthenware body decreases with the temperature of firing is made. Details of the method of determining solubility are given. The results are not concordant, and do not justify the hypothesis. A. T. GREEN.

Dissolution of fused aluminous materials. R. W. ELLISON (J. Amer. Ceram. Soc., 1927, 10, 463).—A method of analysis of fused aluminous products, such as silicates of the sillimanite and mullite series, based on a combination of a bisulphate and a sodium carbonate fusion, is described. A. T. GREEN.

Rumanian kaolins. A. I. BRANISKI (Bul. Chim. Soc. Românâ Stiin., 1926, 29, 15—77).—The Rumanian kaolins have been analysed and classified. None is of markedly superior quality, and many will find application only in the manufacture of stoneware and refractory materials. A few, however, can be used in the manufacture of porcelain, although of a variety inferior to and less translucent than that of Sèvres.

A. B. MANNING.

Properties of refractories in zinc metallurgy.

E. S. WHEELER, A. H. KUECHLER, and H. M. LAWRENCE (Bull. School Mines Met., Miss., 1927, 10, [2], 1—139).—For the manufacture of retorts and condensers for zinc distillation the use of reclaimed retort material as part of the grog is of doubtful economy, but old bricks may be used if they were originally made of a good refractory clay, and the addition of a quantity of properly calcined flint clay is recommended. Diaspore clay is the best grog material, but is usually too expensive. Burley clay is probably the second-best clay material, as bodies made from this clay show less sag and firing shrinkage, and are more resistant to spalling than bodies made from the standard grog of broken saggars. If carborundum fire-sand is used it should have a low iron content, and cyanite must be calcined at 1400° until conversion into mullite is completed. Sagger clays and high-silica grogs usually give less sag and firing shrinkage but a lower modulus of rupture than the standard grog. Tests of various American clays from different localities showed relatively little difference, indicating that the nature of the grog is of more importance than that of the clay. The bulletin contains numerous tables showing the composition and properties of American clays and grogs, and graphs showing the variation of the physical properties of the refractory according to its composition.

A. R. POWELL.

Porosity and permeability. HOWE and HUDSON.—See I.

PATENTS.

Manufacture of hollow bodies from quartz and similar materials. W. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 280,291, 12.8.26).—Hollow bodies closed at one end are made by the electric fusion of quartz etc. by using a hollow resistor closed at one end and having both terminals at the other end, one being connected to the body of the resistor and the other to a tubular internal stem leading to the closed end. The resistor may be spiral, so that heat is radiated through the intermediate spaces.

W. G. CAREY.

Furnace [cement kiln] lining. A. G. CROLL, Assr. to ATLAS PORTLAND CEMENT Co. (U.S.P. 1,647,083, 25.10.27. Appl., 5.7.23).—A rotary cement kiln is lined with bricks of which the outer faces are composed of heat-insulating material, *e.g.*, a mixture of cement and clinker, and the inner faces of a mixture of cement and a heat-conducting material, *e.g.*, iron. Preferably, the latter mixture is formed in layers, in which the percentage of iron increases as the inner surface is approached.

T. S. WHEELER.

IX.—BUILDING MATERIALS.

X-Ray study of magnesium oxychloride cement. T. MAEDA (Sci. Papers Inst. Phys. Chem. Res. Tokyo, 1926, 5, 95—102).—An X-ray study of powdered magnesium oxychloride cements shows the presence of crystalline $3\text{MgO} \cdot \text{MgCl}_2 \cdot 12\text{H}_2\text{O}$ in the cement after setting. Magnesium hydroxide together with magnesium chloride solution yields a product in which definite crystalline structure could not be found, although it appears possible that imperfect crystals might be present. The diffraction lines of magnesium oxide

cannot be found in the diffraction diagram of hardened cement. The presence of crystals in the cement is not, however, a confirmation of Le Chatelier's theory (Trans. Faraday Soc., 1919, 14, 8), as these crystals may be of the dimensions of colloidal particles. R. TRUSZKOWSKI.

Reactions taking place after treatment of calcareous [building] stones with sodium silicate. P. GAVELLE (Chim. et Ind., 1927, 18, 564—585).—Commercial sodium silicate solutions (*d* 1.32) containing 33% of solid silicate ($\text{Na}_2\text{O} \cdot 3 \cdot 5\text{SiO}_2$) behave on dialysis and in diffusion tests as true colloidal solutions, practically no separation of silica and soda taking place in either test. On mixing such solutions with precipitated calcium carbonate and exposing the mixture in thin layers to the air a rapid loss in weight occurs during the first few days due to evaporation of water, but subsequent loss is slight and the total loss is always 46.6% calculated on the weight of silicate solution. The first apparent action is the formation of a gel, followed after prolonged keeping by the appearance of small needle-shaped crystals of hydrated sodium metasilicate. Even after the lapse of a year no appreciable change takes place in the calcium carbonate particles. After a short period of exposure, addition of water regenerates the original colloidal solution, but after longer periods an irreversible gel is formed. In the presence of carbon dioxide the separation of the gel is accelerated. From these results the mechanism of the silicification of calcareous building stones after treatment with sodium silicate is explained as follows. The solution dries on the surface with the formation of a film of gel which slowly becomes irreversible, whilst crystals of hydrated sodium metasilicate form in the interior. The outer layers are slowly decomposed by the carbon dioxide of the air with the deposition of amorphous silica, the sodium carbonate simultaneously formed being washed away by rain. The solution which has penetrated into the pores of the stone is slowly decomposed by the impurities present, calcium carbonate itself being inert, and insoluble double silicates are thus formed in the pores, thereby strengthening the resistance of the stone to further atmospheric denudation.

A. R. POWELL.

Schwabian shale. NEUBRONNER.—See II. **Steam in the lime kiln.** BERGER.—See VII.

PATENT.

Manufacture of a [wood] preservative. R. H. SCHUMACHER, Assr. to B. W. and E. W. FULGHUM, and W. R. SADLER (U.S.P. 1,647,937, 1.11.27. Appl., 15.2.23).—The product obtained by the action of alkali on an oil containing free fatty acids, *e.g.*, fish oil, is mixed with creosote and copper oleate.

T. S. WHEELER.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Apold-Fleissner process of roasting [iron ores in shaft furnaces]. R. BRANHOFFER (Stahl u. Eisen, 1927, 47, 2061—2066).—In the Apold-Fleissner method of roasting spathic iron ore, the ore is passed downwards in a continuous stream through a shaft furnace, through which is passed a current of hot air or flue gas with a low content of carbon dioxide, while a current of cold air passes upwards through the lower part of the shaft

below the hot-air tuyères, this part acting as a cooling chamber for the ore and a preheating flue for the air which rapidly and completely oxidises the ferrous oxide formed in the upper zones of the furnace. The quantity and temperature of the hot gases and cold air used are carefully regulated so as to keep the carbon dioxide content of the flue gas at a minimum and thereby ensure a thorough roasting of the ore at the lowest possible temperature. Diagrams of the furnace used and calculations of the heat balance are given. It is claimed that a furnace roasting 200–450 tons/day requires about 160,000–200,000 kg.-cal./ton with a heat efficiency of 73%.

A. R. POWELL.

Blast-furnace gas. J. F. BARKLEY (U.S. Bur. Mines Tech. Paper, 1927, No. 401, 1–9).—A record of actual and calculated yields of gas per ton of iron.

CHEMICAL ABSTRACTS.

Problem of sulphur in cast iron and steel. J. CIOCHINA (Chim. et Ind., 1927, 18, 600–604).—In striking a balance between the input of sulphur into the blast furnace in the ore, fuel, and fluxes and the output in flue gas, slag, and metal about 50% of the input remains to be accounted for. Evidence is adduced to show that this escapes from the stream of metal during casting, and it is suggested that it volatilises as free sulphur produced by the thermal dissociation of sulphides in the metal stream.

A. R. POWELL.

Calcium carbide as an agent for removing sulphur and phosphorus from iron and steel. W. J. FARRELL, JUN. (J. Western Soc. Eng., 1927, 32, 206–218).—Calcium carbide removes sulphur under reducing conditions and phosphorus during oxidising conditions. The iron is preferably treated with calcium carbide in the hot furnace and poured over a surface covered with the carbide.

CHEMICAL ABSTRACTS.

Desulphurising action of manganese in iron. C. H. HERTY, JUN., and J. M. GAINES, JUN. (U.S. Bur. Mines Rep. Invest., 1927, No. 2817, 8 pp.).—The concentration of manganese and sulphur is expressed by an equilateral hyperbola. The relation between the temperature and the solubility product ($\% \text{ Mn} \times \% \text{ S}$) for manganous sulphide in iron in contact with solid manganous sulphide is shown at 1100–1440° and 1–1.75% Mn. In this range the dissolved manganous sulphide increases rapidly from 0.03 to 0.65%. Probably manganese cannot act in the blast furnace and in the open hearth directly as a desulphuriser.

CHEMICAL ABSTRACTS.

Von Seth's method of extracting vanadium from pig iron. R. VON SETH and G. HULT (Tek. Tid., Uppl. C [Bergsvetenskap], 1927, 57, 20–22).—A description of works' practice for the removal of vanadium and its recovery from the slag. The latter, after grinding and magnetic separation of iron particles, is roasted for 5–8 hrs. at 750–825° with 20% of sodium chloride and a little lime, whereby 75–80% of the vanadium is converted into soluble sodium metavanadate.

CHEMICAL ABSTRACTS.

Iron blast-furnace reactions. S. P. KINNEY, P. H. ROYSTER, and T. L. JOSEPH (U.S. Bur. Mines Tech. Paper, 1927, No. 391, 1–65).—The results of gas-sampling in six planes indicate that the combustion of

coke takes place in a restricted zone at the nose of each tuyère; the extent of the combustion zone controls movement in the upper part of the furnace. The higher carbon dioxide content near the wall indicates greater reduction there in unit area and time.

CHEMICAL ABSTRACTS.

Determination of manganese in steels and other alloys with a high content of chromium or cobalt. E. ROUSSEAU (Chim. et Ind., 1927, 18, 772–780).—The alloy (0.2 g.) is dissolved in 15 c.c. of sulphuric acid diluted with 30 c.c. of water, and organic material derived from the carbides present is oxidised with 3 c.c. of a cold saturated solution of ammonium persulphate. The liquid is neutralised with ammonia, heated to 80° with a further 3 c.c. of persulphate solution, and poured into 40–50 c.c. of ammonia solution. After boiling for 3 min. the liquid is filtered and the precipitate, containing all the iron and manganese, but free from chromium and cobalt, is dissolved in dilute sulphuric acid; the manganese is then determined colorimetrically after oxidation with persulphate in the presence of silver nitrate.

A. R. POWELL.

Colorimetric determination of molybdenum in steel. J. KASSLER (Chem.-Ztg., 1927, 51, 953–954).—A sample (1 g.) of steel drillings is dissolved in 50 c.c. of hydrochloric acid (d 1.12) containing 60 g./litre of sodium phosphate, the solution is oxidised with 25 c.c. of 5% potassium chlorate solution, boiled to expel excess chlorine, diluted with 100 c.c. of hot water, and neutralised with sodium hydroxide, and the vanadic acid reduced with 3 g. of ferrous chloride. The hot solution is then poured in a thin stream into 100 c.c. of 16% sodium hydroxide solution, and the mixture is cooled and diluted to 500 c.c. in a graduated flask; 20 c.c. are filtered through a dry paper, treated with 10 c.c. of 10% potassium thiocyanate solution and 10 c.c. of stannous chloride in hydrochloric acid, and the resulting brown colour compared with that of a standard prepared from pure molybdenum trioxide dissolved in sodium hydroxide and treated in a similar manner. The stannous chloride solution is made by dissolving 30 g. of the salt in 150 c.c. of hot, concentrated hydrochloric acid, diluting to 500 c.c. with acid of d 1.12, and adding 500 c.c. of water.

A. R. POWELL.

Etching agent for determining sulphide inclusions in technical iron. M. KÜNKELE (Werkstoffaussch. Ver. deut. Eisenh. Ber., 1925, No. 75, 3 pp.).—A few drops of a reagent, prepared by dissolving gelatin (5 g.) in water (20 c.c.), adding glycerol (20 c.c.) and sulphuric acid (2 c.c.), and, after mixing, 0.8 g. of silver nitrate in aqueous solution, are spread on the polished surface. The blackened surface is rinsed with water, the thin film of silver rubbed off under water, and the specimen dried with alcohol. The liberated hydrogen sulphide precipitates silver sulphide, which protects the sulphide inclusion and its neighbourhood from further action.

CHEMICAL ABSTRACTS.

Binary systems silicon-iron, iron-phosphorus, iron-manganese. H. ESSER and P. OBERHOFFER (Werkstoffaussch. Ver. deut. Eisenh. Ber., 1925, No. 69, 7 pp.).—With increase in the silicon, phosphorus, and manganese content, the temperature of the A3 trans-

formation rises, whilst the intensity decreases proportionally with the silicon content. Below 2.4% Si (0.4% P) at 1100°, the α - γ change cannot be observed; the temperature of the γ - δ change falls with increase of phosphorus. Up to 1% P the temperature of the A2 change is almost constant; it falls to 1.9%, and is constant at 740° to 13%.

CHEMICAL ABSTRACTS.

Rapid determination of iron in brass. A. ZIMMERMANN (Chem.-Ztg., 1927, 51, 915).—The drillings (2 g.) are dissolved in 10 c.c. of nitric acid (d 1.4), and the solution is evaporated with 10 c.c. of 50% sulphuric acid until copious white fumes are evolved. After addition of 40 c.c. of water and warming until the salts are dissolved, the solution is heated with a zinc rod for 30 min. on the water-bath to reduce the iron to ferrous sulphate and precipitate copper, tin, and lead as sponge metals, decanted from the latter, and titrated with permanganate.

A. R. POWELL.

Aluminium alloys and sea-water corrosion. K. L. MEISSNER (Metallurgist, 1927, 143, 71–74).—Corrosion of "lantal" (cf. B., 1927, 168) and duralumin by the oxidising sodium chloride method increases rapidly with ageing temperature (up to 200° for 16–24 hrs.), and is greater for longer ageing.

CHEMICAL ABSTRACTS.

[Aluminium wires for] electrical transmission lines. VON ZEERLEDER and M. BOSSHARD (Z. Metallk., 1927, 19, 459–470).—The effect of heat-treatment on most of the common aluminium alloys with a high content of aluminium is usually to increase the tensile strength at the expense of the electrical conductivity so that, although the wires obtained are strong enough for use as overhead transmission lines, their conductivity is so much reduced that overheating is likely to occur. Ageing at the ordinary or at elevated temperatures has also a deleterious effect on the electrical conductivity. With alloys containing over 98% Al and small quantities of copper or magnesium together with the usual iron and silicon, satisfactory electrical properties are obtained by annealing for some hours at 160–180°. Thus, after annealing the alloy Aldrey (cf. Fuchs, B., 1927, 846) at 160° for 10 hrs. a conductivity of 33 m./ohm-mm.² is obtained with a tensile strength of 34 kg./mm.², compared with 37.5 m./ohm-mm.² and 7–10 kg./mm.² for 99.9% Al.

A. R. POWELL.

Galvanising duralumin. M. HAAS and O. PÖTTKEN (Z. Metallk., 1927, 19, 448–451).—In order to obtain good adhesion of the deposited metal on duralumin it is necessary to remove completely the thin oxide film. This may be effected by pickling in hot 10% sodium hydroxide solution followed by burnishing with finely-ground porcelain until a clean matt surface is obtained. The article is then made the anode for 40–50 sec. in a bath containing 200 g. of ferrous chloride and 175 g. of calcium chloride per litre. Without removing the article from the bath the current is reversed, and in 5–10 sec. a thin coating of iron is deposited which prevents further film formation. For both the preceding operations a current density of 3 amp./dm.² is employed, and the bath is kept at 18–20°. After rinsing the article in water it is plated with a thin coating of copper (0.005 mm. thick) from an acid or

cyanide bath, and finally plated with zinc, nickel, cadmium, or chromium as required. The acid copper bath comprises a solution of 180 g. of copper sulphate crystals, 60 g. of sulphuric acid, and 2 g. of gelatin in 1 litre of water, and is operated at 0.4–0.5 amp./dm.² at 20°. The cyanide bath contains 14 g. of copper as potassium cupricyanide and 10 g. of free cyanide per litre; the best results are obtained at 40° with a current density of 0.4 amp./dm.², the bath being agitated during use. In either case the deposit obtained in 30 min. is sufficient to resist penetration by 10% sodium hydroxide solution for at least 15 min.

A. R. POWELL.

Constitution of magnesium alloys. W. SCHMIDT (Z. Metallk., 1927, 19, 452–455).—Magnesium and silicon form a single compound Mg_2Si , which forms a eutectic with magnesium containing 1.4% Si (m.p. 645°); the compound does not enter into solid solution in magnesium, so that as little as 0.1% Si in magnesium may be detected in the microstructure by the presence of the eutectic along the grain boundaries. The saturated solid solution of aluminium in magnesium contains 7.5% Al at temperatures below 300°; at higher temperatures the solubility increases to a maximum of 11% Al at the eutectic temperature, 436°, hence alloys containing 7.5–11% Al show age-hardening phenomena. The solubility of $MgZn_2$ in solid magnesium is equivalent to 1.8% Zn between 0° and 150°, then increases rapidly to the equivalent of 6% Zn at the eutectic temperature, 344°; alloys containing 1.8–6% Zn are therefore hardened by quenching from 325° and ageing at 100°. Magnesium retains 18% Pb in solid solution at temperatures up to 300° and 26% Pb at the eutectic temperature, 459°. Manganese forms no solid solution or eutectic with magnesium, but a compound of uncertain composition appears to be present in alloys containing over 95% Mg.

A. R. POWELL.

Reduction of metallic oxides by hydrogen. III. Nickel oxide. IV. Cobalt oxide. G. GALLO (Annali Chim. Appl., 1927, 17, 535–543, 544–552. Cf. B., 1927, 278, 279).—III. The reduction of nickel sesquioxide to metallic nickel by hydrogen proceeds continuously, the green oxide, NiO , being formed as an intermediate product. The first stage, $Ni_2O_3 + H_2 \rightarrow 2NiO + H_2O$, begins at 92–95°, at which temperature the dissociation pressure attains an appreciable magnitude, whilst reduction of the green oxide requires the temperature 118–120°. In presence of water vapour of pressure 20 (92) mm., reduction of the sesquioxide commences at 120–122° (130–135°) and requires longer for its completion than by the dry gas. The difference between these results and those obtained by Glaser (B., 1903, 1051) rests on the facts that an appreciable amount of water vapour would be necessary to change the colour of the anhydrous cobalt chloride used by this author to detect the commencement of the reduction, and that part of the water vapour would, doubtless, escape absorption by the indicator.

IV. In presence of pure hydrogen, the dissociation pressure of the oxides Co_2O_3 and Co_4O_5 becomes appreciable, indicative of incipient reduction, at 80–85°. When the reduction is continued at a higher temperature, the oxide Co_3O_4 is apparently formed transitorily and

begins to undergo reduction at about 90° to form the more stable CoO. Reduction of the latter appears to commence at 118–120°, but metallic cobalt is obtained only after 4 hrs. at 400°, 3 hrs. at 500°, 1 hr. at 600°, or 15 min. at 700°; the oxide Co₂O does not form an intermediate step in this reduction. In two instances, reduction of the sesquioxide was accompanied by the appearance, at 200–250°, of a characteristic pink unstable mass of the composition Co₄O₅, although the ordinary oxide of this formula is black and stable. In presence of water vapour of pressure 20 (92) mm., reduction of cobalt sesquioxide by hydrogen begins at about 95° (110°).

T. H. POPE.

Corrodibility of metals used for [cooking] utensils. F. SPRINKMEYER (Z. Unters. Lebensm., 1927, 54, 216–227).—A number of metals and alloys have been examined to determine the extent to which food-stuffs are liable to be contaminated by such metals as lead, zinc, nickel, and copper. Spoons made from the metals under investigation were partially immersed in acetic acid (3.5–4.0%), and in dilute commercial vinegar; in some cases sodium chloride was added. A definite area of each spoon was exposed to the acid at temperatures of 15°, 50°, and 100°, and for times varying from $\frac{1}{4}$ hr. to 24 hrs., the amounts of metal in solution being subsequently determined. With Britannia metal of different grades and containing from 2 to 30% Pb, the amounts of lead dissolved by the dilute acetic acid were very small, the maximum being 1.37 mg. when 40 cm.² of metal were exposed for 12 hrs. at 17–18°. With diluted vinegar the results were appreciably higher, being 3.8 mg. after 6 hrs. exposure at 17–18° and 4.15 mg. after 1 hr. at 50°. With more dilute acid and with alloys less rich in lead, the amounts dissolved were proportionately smaller. The weight of lead dissolved by an aqueous solution of sodium chloride (3.5%) was never higher than 1.0 mg. With a mixture of 0.5% tartaric acid and 0.5% acetic acid at 17–18°, 0.72 mg. of lead was dissolved in 6 hrs. from the 30% lead alloy, and 0.75 mg. in 1 hr. at 50°. Similar experiments were conducted with zinc alloys, zinc-coated brass, and German-silver utensils. Considerable amounts of zinc were dissolved in some cases. With an alloy containing 92% Zn, after immersion for 24 hrs. at ordinary temperatures in a solution containing 3.5% of acetic acid and 0.5% of sodium chloride, 3006 mg. of zinc were found in solution, whilst with a 65% zinc alloy 2009 mg. were found. At higher temperatures the amounts dissolved were considerably increased. With brass and German silver there was very little zinc or copper dissolved at any temperature. The corrosion of the metals was apparent from the changed appearance of the spoons and from the scum which formed on the liquid. The action of a weakly alkaline mineral water was also investigated, but only insignificant amounts of zinc were dissolved. The author urges that the manufacture and sale of utensils made from zinc or alloys containing more than 45% Zn should be prohibited.

H. J. DOWDEN.

Granulation of slags. B. BOGITCH (Rev. Mét., 1927, 24, 695).—To avoid risk of explosion when granulating slags, the stream of slag should be at such a temperature that it flows freely from the furnace into

water preheated at 35–40°. Care should be taken that no slag runs down the walls into the side of the granulation tank, as this almost invariably gives rise to explosions.

A. R. POWELL.

Magnesium and its alloys. I, II. S. L. ARCHBUTT (Metallurgist, 1926, 141, 4–6).

Refractories. WHEELER and others.—See VIII.

PATENTS.

Furnace with blast heater. E. VANDERSTEIN (U.S.P. 1,649,201, 15.11.27. Appl., 21.1.25. Belg., 29.10.24).—A pair of blast furnaces have retort chambers at opposite sides of each furnace, these chambers being in direct communication with regenerators and preheaters. A reversing valve control box is so arranged that, when the air supply communicates with the regenerating system of one furnace, that of the other furnace communicates with the exhaust, and *vice versa*.

C. O. HARVEY.

Reverberatory furnace. G. W. PRINCE, ASSR. to UNITED VERDE EXTENSION MINING Co. (U.S.P. 1,650,907, 29.11.27. Appl., 23.7.26).—Fettling material is supplied, at the firing end of the furnace chamber, to the side walls thereof, to protect them from the action of the smelting ore and the intense heat of the chamber.

II. ROYAL-DAWSON.

Production of cast iron of low carbon content in cupola furnaces. SOC. ALSACIENNE DE CONSTRUCTIONS MÉCANIQUES (F.P. 618,597, 17.11.25).—Steel scrap in small pieces, mixed with predetermined quantities of silicon and manganese, is melted rapidly in a cupola. This is effected by using a larger proportion of coke than usual, enlarging the cross-section of the tuyères, and increasing the pressure of the air blast.

A. R. POWELL.

Preventing the diffusion of carbon into iron during cementation. A. LENNARTZ (G.P. 444,126, 16.2.26).—To prevent diffusion of carbon into certain parts of an iron or steel article during cementation of the remainder, the parts are covered with a mixture of kaolin or the like and a sulphur compound non-volatile below 850°, e.g., ferrous sulphide.

A. R. POWELL.

Refining of iron. G. S. EVANS, ASSR. to MATHIESON ALKALI WORKS (U.S.P. 1,646,728, 25.10.27. Appl., 7.6.26).—Molten iron is led through an elongated passage in which it is thoroughly mixed with soda ash, and thence into a receptacle in which the slag formed is separated.

T. S. WHEELER.

Steel alloy. D. J. GILES, ASSR. to LATROBE ELECTRIC STEEL Co. (U.S.P. 1,650,707, 29.11.27. Appl., 2.4.26).—An alloy which is highly resistant to attack in either its annealed or hardened state contains 17.00% Cr and 0.60% C, the remainder being iron, together with the usual amounts of other elements which enter during its manufacture.

F. G. CROSSE.

Production of corrosion-resisting coatings on iron and steel products. C. H. HUMPHRIES, ASSR. to METALS PROTECTION CORP. (U.S.P. 1,651,278, 29.11.27. Appl., 5.6.24).—The surfaces are plated first with copper and then with chromium.

F. G. CROSSE.

Corrosion-resisting alloy. P. D. SCHENCK, ASSR. to DURIRON Co., INC. (U.S.P. 1,651,970, 6.12.27. Appl.,

11.8.25).—The alloy contains approximately 89% Cu, 7% Al, 3% Fe, and 1% Ni. F. G. CROSSE.

Manufacture of welded high-pressure vessels from vanadium steel. R. FRENZEL (G.P. 443,999, 20.9.25).—Vessels of several metres in diameter and 12–15 m. in length, resistant to pressure at moderately high temperatures, are constructed of ingot steel with a low carbon content and containing 0.15–0.25% V. L. A. COLES.

Recovery of copper and nickel from solutions and residues. E. ABEL (G.P. 443,910, 28.3.23. Austr., 8.4.22).—Residues containing copper and nickel are heated in an atmosphere of hydrogen chloride with only a limited supply of air so that the copper is converted mainly into cuprous chloride. The produce is leached with hot dilute hydrochloric acid so that the solution, after cooling, is saturated with respect to both cuprous and nickel chlorides. The insoluble portion is leached with a strong solution of a chloride to dissolve the cuprous chloride, and the solution of the soluble portion is treated with nickel scrap to remove the remainder of the copper. A. R. POWELL.

Recovery of copper, lead, zinc, and silver from low-grade calcareous ores. V. TAFEL and W. WITTER (G.P. 444,612, 24.7.25).—The ore is given a chloridising roast at a sufficiently high temperature to effect complete volatilisation of the chlorides of copper, lead, zinc, and silver. A. R. POWELL.

Production of silicon-iron and aluminium-copper alloys. F. D. SHUMAKER, ASST. to ALUMINUM CO. OF AMERICA (U.S.P. 1,644,000, 4.10.27. Appl., 7.3.23).—The application of the process described in E.P. 208,716 (B., 1925, 508) is facilitated by the addition of magnesium oxide in the primary smelting operation. T. S. WHEELER.

Purification of beryllium. SIEMENS & HALSKE A.-G., ASSECS. of H. FISCHER (G.P. 443,944, 11.9.25).—Compact beryllium metal is immersed in molten mixtures of alkali chlorides containing lithium chloride, whereby the outer layer of impurities is dissolved in a few minutes. The metal is removed from the bath, washed, dried, and burnished lightly. A. R. POWELL.

Production of pure magnesium. A. JESSUP (F.P. 618,875, 20.11.25).—A solution of magnesia in molten magnesium fluoride is electrolysed using as cathode lead, tin, or copper silicide so as to obtain a magnesium alloy, which is then heated more strongly in order to volatilise the magnesium. A. R. POWELL.

Solder for aluminium and its alloys. A. PASSALACQUA (Addn. No. 31,454, 27.7.25, to F.P. 611,114; B., 1927, 969).—A solder for aluminium consists of tin, antimony, zinc, aluminium, magnesium, lead, sodium, phosphorus, and copper in suitable proportions. A. R. POWELL.

Manufacture of liquid zinc from blue powder. F. THARALDSEN (U.S.P. 1,647,381, 1.11.27. Appl., 21.11.23. Norw., 28.11.22).—Zinc dust is melted in a rotary kiln, the inner surface of which is fitted with scoops for raising the molten metal and pouring it on to the powder. T. S. WHEELER.

Recovery of zinc oxide [from ores]. C. A. H. DE SAULLES, ASST. to AMER. SMELTING & REFINING CO.

(U.S.P. 1,647,279, 1.11.27. Appl., 31.12.21. Renewed 29.3.27).—Zinc ore mixed with a reducing agent is delivered on to a sloping embankment in a reverberatory furnace, down which it passes in a thin layer to the hearth. The zinc present is completely volatilised before the material reaches the hearth, and is oxidised in the combustion space above the furnace. T. S. WHEELER.

Metallurgical method [preparation of thorium etc.]. J. W. MARDEN, ASST. to WESTINGHOUSE LAMP CO. (U.S.P. 1,646,734, 25.10.27. Appl., 21.8.22).—Thorium, uranium, zirconium, titanium, vanadium, tantalum, or tungsten chloride is heated in a closed vessel out of contact with oxygen at a red heat for 1 hr. with calcium chloride and sodium to yield a mixture of the corresponding metal, sodium chloride, and calcium oxide, which is treated with alcohol to remove residual sodium, and with dilute acetic acid. Iron is extracted magnetically, and the residue is finally washed with alcoholic hydrogen chloride solution to leave the pure metal corresponding to the halide treated, e.g., thorium. T. S. WHEELER.

[Solder for] tungsten welding. C. W. BALKE, ASST. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,651,662, 6.12.27. Appl., 24.11.26).—A solder for floating tungsten contact discs on nickel breaker arms contains, approximately, 60% Ag, 15% Cu, 5% Ni, and 20% Zn. F. G. CROSSE.

[Brazing] solder. J. L. JONES, ASST. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,651,709, 6.12.27. Appl., 30.6.21).—An alloy consisting of 6–10% P and the remainder copper is claimed. F. G. CROSSE.

Recovery of platinum metals from ores. W. GÜNTHER (G.P. 444,219, 8.5.25).—The ore is treated with carbonyl chloride under pressure to convert the platinum metals into carbonyl derivatives of their chlorides, which may be recovered by leaching the treated ore with suitable solvents or by heating it under diminished pressure to volatilise these compounds. A. R. POWELL.

Borating of Dumet wire. R. D. HALL, ASST. to WESTINGHOUSE LAMP CO. (U.S.P. 1,647,620, 1.11.27. Appl., 20.2.26).—The process of E.P. 4235 of 1914 (B., 1915, 347) is modified in that the wire while hot is maintained out of contact with oxygen. T. S. WHEELER.

Decomposition of chromium ores and production of electrodeposited chromium. E. LIEBREICH (G.P. 443,200, 17.7.24; cf. B.P. 243,046; B., 1926, 96).—Chromium ores are oxidised at the ordinary temperature or by heating to obtain chromium chromate, and solutions of this compound are used for chromium plating baths, the simultaneous presence of iron being without deleterious action. A. R. POWELL.

Manufacture of a flotation agent. B. L. GEORGE (U.S.P. 1,647,793, 1.11.27. Appl., 2.2.25).—Coal-tar oil is treated at 100–120° with sodium hydroxide (0.25%) and sulphur (0.5%) to render it of value as a flotation agent for ores. T. S. WHEELER.

Flotation method and apparatus [for concentration of ores]. D. L. FORRESTER (U.S.P. 1,646,019, 18.10.27. Appl., 29.7.25).—A pulp of the ore is aerated by large air bubbles discharged into it well below the surface, co-acting currents of the pulp being directed against the bubbles near their point of entry. The pulp

is caused to circulate through a zone of violent agitation, produced by the air, and a quiescent zone. Both zones are under normal atmospheric pressure and isolated from each other, the flow from the first to the second being confined to a restricted horizontal path beneath the surface of the pulp. Mineral-bearing froth is removed from the quiescent zone. H. HOLMES.

Gold separator. S. NILES (U.S.P. 1,646,309, 18.10.27. Appl., 2.7.25).—A hopper is mounted over a tank with its lower end forming a casing which terminates close to the tank floor. The lower end of a helical conveyor extending through the hopper and the casing carries spreader blades extending between, and fitting against, the end of the casing and the tank floor. Small openings are provided between the blades to enable a small amount of material to be fed from the lower end of the casing to a point beneath the blades.

H. HOLMES.

Treatment of metal articles. M. BENTLEY, Assr. to PACKARD MOTOR CAR CO. (U.S.P. 1,647,851, 1.11.27. Appl., 13.7.23).—The forging to receive a Babbitt-metal bearing is first washed to remove dirt, then coated on the surface with basic iron phosphate, the bearing portion machined, and finally dipped in a tinning bath before making the casting. H. ROYAL-DAWSON.

Smelting furnace. F. W. CORSALLI (U.S.P. 1,647,608, 1.11.27. Appl., 9.8.21. Ger., 19.9.13).—A smelting furnace comprises a flame-producing apparatus arranged to direct flame into the main part of the furnace and to heat an auxiliary part, a slag chamber to receive slag and waste flame, heat-accumulating chambers connected to the slag chamber and heated by waste flame, and means for directing the waste flame continuously in one direction in order to maintain the slag in the liquid state in the slag chamber. J. S. G. THOMAS.

Lithium-containing bearing metals. G. WELTER, Assr. to ALLIED PROCESS CORP. (U.S.P. 1,652,077, 6.12.27. Appl., 6.3.25. Ger., 12.3.24).—See B.P. 230,827; B., 1925, 767.

Lead-alkali metal-containing bearing metal alloys. G. WELTER, Assr. to ALLIED PROCESS CORP. (U.S.P. 1,652,078, 6.12.27. Appl., 13.7.25. Ger., 24.7.24).—See B.P. 237,583; B., 1925, 854.

Manufacture of very hard metallic alloys. H. LOHMANN (U.S.P. 1,652,027, 6.12.27. Appl., 26.8.21. Ger., 7.12.18).—See B.P. 157,774; B., 1922, 470 A.

Froth flotation apparatus (G.P. 443,616).—See I.

XI.—ELECTROTECHNICS.

Operation and regulation of electric furnaces studied diagrammatically. P. BERGEON (Rev. Mét., 1927, 24, 683—694).—Mathematical. Diagrams and formulae are given illustrating the dependence of the power factor on the electrical conditions and method of operating an electric furnace operated from a transformer. The results obtained suggest a new method of regulating arc furnaces by suitable manipulation of the electrodes which is especially advantageous when the voltage of the primary is adjustable and the furnace has a relatively high inductance. A. R. POWELL.

PATENTS.

Magnetic [dust] core. Insulation of magnetic material. V. E. LEGG, Assr. to BELL TELEPHONE LABORATORIES, INC. (U.S.P. 1,647,737—8, 1.11.27. Appl., [A] 3.1.27, [B] 5.1.27).—(A) A finely-divided mixture of nickel-iron alloy and lead sesquioxide (5%) is formed and pressed, and then heated at 500° to improve the magnetic qualities of the alloy and produce lead monoxide together with an insulating layer of oxide on the alloy particles. (B) The powdered alloy, after being heated in air to form a surface layer of oxide and mixed with quartz powder, is formed and pressed, so that quartz enters the pores of the oxide layer and improves the insulation of the alloy particles. T. S. WHEELER.

Chemical treatment of dielectric material [mineral oil, turpentine, etc.] in an alternating electric field. SIEMENS & HALSKE A.-G., Assces. of H. GERDIEN (G.P. 444,108, 25.5.21).—The material is brought in contact with inert gases while it flows over porous electrodes also constructed of dielectric material.

L. A. COLES.

[Preparation of tungsten filaments for electric] incandescence lamps. G. R. FONDA, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,648,679, 8.11.27. Appl., 18.1.21).—A mixture of finely-powdered tungsten and less than 1% by wt. of boron is consolidated and heat-treated, and the metal mechanically rendered ductile. The resulting metal is finer grained, of lower vapour pressure, and exhibits less sagging at high temperatures than tungsten unadmixed with boron. J. S. G. THOMAS.

Incandescence electric lamp. J. H. DE BOER, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,648,677, 8.11.27. Appl., 2.11.26. Holl., 12.12.25).—A tungsten filament is sealed in a bulb containing a salt of hydrofluoboric acid to reduce absorption of light by vaporized tungsten. J. S. G. THOMAS.

Thermocouple. O. HERMANN, Assr. to THERMO-ELECTRIC BATTERY CO. (U.S.P. 1,648,231, 8.11.27. Appl., 9.11.25. Renewed 28.8.26).—In a thermocouple, the positive element consists of an alloy containing 44—46% Ni, 1—3% Co, and 53% Cu, and the negative element of an alloy containing 83—83.5% Ni and 16.5—17% Mo. F. G. CROSSE.

Production of a high vacuum in vessels [electric lamps]. D. S. GUSTIN, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,647,618, 1.11.27. Appl., 6.9.22).—A vessel to be evacuated is filled with a vapour only, in which a physical change is effected by passing an electric discharge between external and internal electrodes. J. S. G. THOMAS.

Apparatus for measuring ionic concentration. E. MISLOWITZER (U.S.P. 1,648,739, 8.11.27. Appl., 7.11.25. Ger., 31.1.25).—Apparatus for measuring ionic concentration comprises outer and inner vessels for holding liquid each provided with an electrode and a ground annular surface. An engaging top part includes inner and outer coaxial cylinders closed at the upper ends to form an inverted annular container and having ground annular surfaces at the lower ends of both cylinders, and is adapted for the insertion of an electrode and for filling purposes. J. S. G. THOMAS.

Conducting [electric] currents. K. H. KINGDON and I. LANGMUIR, ASSES. to GEN. ELECTRIC CO. (U.S.P. 1,648,183, 8.11.27. Appl., 21.12.22).—Contact is made between a positively charged electrode and the vapour of a substance which is chemically inert towards the electrode and has a lower electron affinity. The electrode is heated above a critical temperature and the positive ions generated are removed by an applied potential.

J. S. G. THOMAS.

Electrolytic rectifier. W. MORRISON (U.S.P. 1,647,649 and 1,647,650, 1.11.27. Appl., 15.10.25).—A filming electrode of niobium or tantalum and a non-filming electrode of lead, platinum, or carbon in a solution of (A) zirconium sulphate, or (B) a rare-earth metal sulphate, are employed.

T. S. WHEELER.

Magnesium (F.P. 618,875). **Chromium** (G.P. 443,200).—See X.

Purification of sugar juices (B.P. 280,321).—See XVII.

XII.—FATS; OILS; WAXES.

Bromine-iodine value of butter and other edible fats. W. VAUBEL (Z. Unters. Lebensm., 1927, 54, 275—279).—Primary bromine-iodine values correspond closely with iodine values; secondary and tertiary values are higher. Saponified summer butter has a decidedly lower bromine-iodine value than have unsaponified samples. The reverse is the case with the majority of winter butters. Values for winter butters are generally lower than those for summer butters. The bromine-iodine value for butters includes bromine absorbed by unaltered fatty acids present, by molecules formed by the union of two fatty acid molecules in a tetramethylene form of combination, by acids of the linoleic type, and by trimethylamine and formaldehyde produced by the decomposition of choline. The quantity of choline present is indicated by the difference between the bromine-iodine value of saponified and unsaponified samples.

A. G. POLLARD.

Origin and detection of rancidity in fats and oils. J. PRITZKER and R. JUNGKUNZ (Z. Unters. Lebensm., 1927, 54, 242—252).—Earlier work of the authors (B., 1926, 1020) is discussed in conjunction with that of Powick (cf. A., 1923, ii, 191; B., 1924, 302). The mechanism of the Kreis test for rancidity is further explored.

A. G. POLLARD.

Determination of butter fat and coconut oil. M. KLOSTERMANN and H. QUAST (Z. Unters. Lebensm., 1927, 54, 297—302).—The method of Bertram, Bos, and Verhagen (cf. B., 1926, 499) is lengthy and requires a large sample (25 g.) for each determination. The following simplified method is adopted. A 5 g. sample of fat is saponified with 3 g. of caustic potash solution (1:1) and 8 g. of glycerin. The soap is dissolved in water, and the solution diluted to 100 c.c., warmed to 80°, and the magnesium soap precipitated by means of 25 c.c. of a cold magnesium sulphate solution (150 g./litre). After filtration a portion of the filtrate is treated with excess of silver nitrate (25 c.c. of 0.1*N*-solution) and the excess determined by Volhard's method. The silver used up represents one half the "A" number. The filtrate from the silver thiocyanate

precipitate is treated with 10% caustic potash solution until precipitation of iron begins, when 1 g. of oxalic acid is added and the liquid distilled. The number of c.c. of 0.1*N*-potassium hydroxide solution required to neutralise the distillate is half the "B" number. Results thus obtained accord with Reichert-Meissl and Polenske values. The effects of caproic, caprylic, capric, lauric, and myristic acids on the process are determined and discussed.

A. G. POLLARD.

Almond, apricot kernel, and peach kernel oils. J. PRITZKER and R. JUNGKUNZ (Z. Unters. Lebensm., 1927, 54, 233—242).—The difficulty of identifying peach and apricot oils as adulterants of almond oil is emphasised. Complete analyses of pure and commercial samples of the three oils are recorded. Bellier's test gives a negative result with pure almond oil. The Kreis reaction for peach kernel oil is characteristic of apricot kernel oil and not of peach oil. A 5% admixture of apricot oil in almond oil can be identified by this means. The Maben reaction is negative for all three oils. Commercial samples of each of the specified oils show analytical figures intermediate between those of the pure oils. Most apricot oil appears commercially as "peach oil," which in a pure condition is not a commercial commodity.

A. G. POLLARD.

Margarine manufacture. SCHEUNERT.—See XIX.

PATENTS.

Treatment of articles with soaps. I. G. FARBENIND. A.-G. (B.P. 253,105, 31.5.26. Ger., 2.6.25).—The separation of fatty acids or insoluble salts, when soaps are used with water containing acid or calcium or magnesium salts, or with salt water, is prevented by addition of sulphite-cellulose waste liquor or a hydro-aromatic or aromatic sulphonie acid of high-wetting power. *E.g.*, diisopropylnaphthalenesulphonic acid is added to an acid dye bath containing soap or to a slightly acid fulling bath.

C. HOLLINS.

Soap preparations and treatment of articles therewith. I. G. FARBENIND. A.-G. (B.P. 280,110, 31.5.26. Addn. to B.P. 253,105, preceding).—The wetting-out agent is here incorporated with the soap prior to use. Purifying agents (borax) or organic solvents (glycerol, benzene, alcohols), and also sulphite-cellulose waste liquor may be added to the mixture.

C. HOLLINS.

Separation of tall oil into its constituents. R. ROLL (G.P. 443,960, 17.6.23).—The oil is saponified in solution in an organic solvent, and the resin soaps formed at first are separated from the fatty acid soaps formed later.

L. A. COLES.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Solubility of lake pigments in cellulose varnishes. H. WAGNER and J. KESSELRING (Farben-Ztg., 1927, 33, 619—621).—The difference in "fastness" of various lake pigments to oil paints and cellulose lacquers is discussed, special attention being given to "bleeding" and to change of colour of mixed pigments. Tables are given summarising information on these points and on the solubility of typical lake pigments in various solvents, diluents, plasticisers, etc.

S. S. WOOLF.

Testing of finished lacquers. J. D. JENKINS and P. R. CROLL (Ind. Eng. Chem., 1927, 19, 1356—1358).—Two classes of tests on lacquer films are described. The accelerated exposure tests, which consist of a cycle of exposure to ultra-violet light, water, and refrigeration, are useful for comparative purposes, but cannot be correlated with normal exposure tests. The tests of physical properties comprise the determination of elongation at rupture, contraction on drying, and ease of sanding. F. R. ENNOS.

Malayan varnish resins. T. H. BARRY (J. Roy. Soc. Arts, 1927, 76, 106—128).

PATENTS.

Production of low-viscosity lacquer and film. E. M. FLAHERTY, Assr. to E. I. DUPONT DE NEMOURS & Co. (Re-issue 16,803, 29.11.27, of U.S.P. 1,629,999, 24.5.27).—See B., 1927, 563.

Indicator for reactions in liquid media (G.P. 443,454).—See I.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Reactions of aniline and its homologues in accelerating vulcanisation. T. KIMISHIMA (Mem. Coll. Eng. Kyushu, 1927, 4, 193—250).—The reaction products of sulphur with aniline, methylaniline, dimethylaniline, and *p*-toluidine are shown to consist essentially of disulphide compounds and hydrogen sulphide. Aniline, for instance, at 160—170°, gives *o*-diaminodiphenyl disulphide and a resinous polysulphide, with smaller proportions of *o*-diaminodiphenyl sulphide and *p*-diaminodiphenyl disulphide. The disulphides are accelerators of vulcanisation, particularly in an atmosphere of hydrogen sulphide, their conversion into the corresponding mercaptan and re-formation by sulphur probably being involved in their method of action. The relative effectiveness of aniline and its homologues is naturally influenced by the properties of the disulphides formed, but the main factor is the quantity of disulphide or polysulphide produced. Any monosulphide formed is relatively ineffective, whilst certain of the products, *e.g.*, methylaminophenyl mercaptan, may even have a retarding action. The thermal changes during the vulcanisation of a mixture of rubber (92½) and sulphur (7½) with a small proportion of various organic accelerators were followed by the method of Williams and Beaver (B., 1923, 412 A), and it is shown that by working at 175° the extent of self-heating provides an index as to the degree of acceleration. From their favourable influence on the tensile strength of the products it is possible that *p*-diaminodiphenyl disulphide and 2:2'-dimethyl-5:5'-diaminodiphenyl disulphide may prove of industrial value. D. F. TWISS.

Relative action of vulcanisation accelerators in vulcanite mixings. R. DITMAR (Chem.-Ztg., 1927, 51, 901—902).—Examination of the behaviour of a series of accelerators in a vulcanite mixing shows that with litharge as activator, the relative results are generally different from those obtained with zinc oxide. The latter activator also tends to cause a volume increase during vulcanisation. With lithopone the relative

results are different from those with either zinc oxide or litharge. D. F. TWISS.

Conversion of rubber into thermoplastic products with properties similar to gutta-percha, balata, and shellac. I. Preparation and general properties. H. L. FISHER. II. Chemistry of the reaction. H. L. FISHER and E. M. MCCOLM (Ind. Eng. Chem., 1927, 19, 1325—1328, 1328—1333).—Tough thermoplastic products resembling gutta-percha can be obtained by heating a sheeted mixture of rubber with approximately 10% of an organic sulphonic acid or sulphonyl chloride, *e.g.*, *p*-toluenesulphonic acid or *p*-toluenesulphonyl chloride at 125—135° for several hours; if the mixture is heated in bulk the product more closely resembles shellac. If heated with 7½% of *p*-phenolsulphonic acid, rubber yields a gutta-like material soluble in benzene and a good adhesive, which is the basis of the Vulcalock process. Sulphuric acid (5%) converts rubber into products of the gutta-type, but not of the soluble shellac type. The general name "thermoprene" with a qualifying suffix is applied to these products, which appear to have the same empirical composition as rubber. The chemical unsaturation of the shellac-like product towards sulphur, Wijs' reagent, or oxidation is only 55—60% of that of rubber. The purified hydrocarbon is colourless, soluble in the ordinary rubber solvents, and more stable than rubber towards heat. D. F. TWISS.

Colloid chemistry of rubber latices. II. Surface tension measurements with latex of *Hevea brasiliensis*. E. A. HAUSER and P. SCHOLZ (Kautschuk, 1927, 332—333).—The results of surface tension measurements for fresh *Hevea* latex, by means of a torsion balance, are compared with those for various soaps at equivalent dilutions. Fresh latex contains some substance with a marked depressive influence on surface tension, its effect being almost as great as that of a normal soap. The value of the surface tension attains a minimum when the latex is diluted to ¼th of its original concentration. The surface tension of ammonia-preserved latex gradually decreases with storage. After coagulation of latex, the substance which depresses surface tension is found in the serum. D. F. TWISS.

Rubber mixings. W. ESCH (Kautschuk, 1927, 337—345).—A consideration of typical mixings for various purposes and a discussion of the choice of ingredients. D. F. TWISS.

Rubber mixings. W. B. WIEGAND (Kautschuk, 1927, 334—336).—The present position with respect to the use of carbon black in rubber and in other directions is reviewed. For mixings containing a high proportion of carbon black it is preferable to choose a vulcanisation accelerator of moderate activity, *e.g.*, one of the aldehyde-aniline type. D. F. TWISS.

PATENTS.

Vulcanising of rubber. N. A. SHEPARD and S. KRALL, Assrs. to FIRESTONE TYRE & RUBBER Co. (U.S.P. 1,647,754, 1.11.27. Appl., 20.6.23. Renewed 5.2.26).—The reaction products of thiocarbamide or thiocarbimides and aldehyde-ammonia compounds are of value as accelerators. T. S. WHEELER.

Production of a [vulcanised] rubber product. S. G. LUTHER (U.S.P. 1,647,184, 1.11.27. Appl., 19.2.27).

—A mixture of a soluble, *e.g.*, sodium, and of an insoluble, *e.g.*, zinc, soap is of value as an accelerator.

T. S. WHEELER.

Treatment of [rubber] latex. J. MCGAVACK, Asst. to NAUGATUCK CHEMICAL CO. (U.S.P. 1,647,805, 1.11.27. Appl., 24.3.26).—Latex is treated with pectin, and the concentrated rubber layer which separates is, after addition of a sodium soap, heated at 80° for 2 hrs. to reduce the viscosity.

T. S. WHEELER.

Manufacture of stabilised [rubber] latex. RUBBER LATEX RES. CORP., Assees. of M. R. DAY (B.P. 279,336, 7.5.27. U.S., 3.1.27).—Rubber latex is stabilised by the addition of from 1% to 10% of blood or of components of blood, especially the hæmoglobin. D. F. TWISS.

Protective linings for [petrol] tanks. SOC. ELECTRO-MECANIQUE D'APPAREILLAGE POUR L'ESSENCE (B.P. 279,291, 28.1.27. Belg., 31.12.26).—For the automatic sealing of shot-holes in the petrol tanks of aircraft the well-known rubber jacket is replaced by one of guayule (or a mixture of this with ordinary rubber), which is less sensitive to temperature changes and is more rapidly attacked by petrol. D. F. TWISS.

Protection of rubber articles against ageing. SOC. ITAL. PIRELLI (B.P. 260,001, 18.10.26. Italy, 17.10.25).—The residue obtained from latex serum by evaporation, preferably under reduced pressure, is mixed with rubber as an antidote against bad ageing. The proportion introduced is equal to, or greater than, that corresponding with the amount associated with the rubber when in the latex. The quebrachitol has no useful effect in this direction and may be removed, together with the proteins, from the concentrated serum. D. F. TWISS.

Providing metal, wood, and like surfaces with a hard rubber-containing coating. R. M. WITTHY-COMBE (B.P. 258,573, 6.9.26. Austral., 18.9.25).—A vulcanisable rubber solution containing asphaltum or similar material is applied once or repeatedly to the surface. The whole is then subjected to dry heat between 120° and 260°. Gradual heating gives a surface with a fine smooth finish, whereas with rapid heating a matt surface is obtained. D. F. TWISS.

Compounded rubber. F. G. BREYER and C. W. FARBER, Assts. to NEW JERSEY ZINC CO. (U.S.P. 1,649,782, 15.11.27. Appl., 27.7.21).—The dried mixture of zinc sulphide and barium sulphate obtained by coprecipitation, but not subsequently calcined, is an effective reinforcing ingredient for rubber. D. F. TWISS.

Concentration of fluids [latex etc.]. W. A. GIBBONS and M. G. SHEPARD, Assts. to GEN. RUBBER CO. (U.S.P. 1,651,764, 6.12.27. Appl., 11.7.23).—See B.P. 218,544; B., 1924, 757.

Manufacture of porous bodies, diaphragms, filters, etc. of ebonite. M. WILDERMANN (U.S.P. 1,651,567, 6.12.27. Appl., 22.4.22. Ger., 1.4.22).—See B.P. 200,577; B., 1923, 899 A.

Manufacture or utilisation of india-rubber. L. C. BATEMAN (B.P. 279,531, 26.6.26).

XV.—LEATHER; GLUE.

Curried upper leathers. Wear of kips, splits, etc., and influence of various dressings. A. COLIN-RUSS (J. Soc. Leather Trades' Chem., 1927, 11, 336—349).—Currying should produce a flexible leather without greasy feel or exudation of greasy matter under mere mechanical action. The currying grease should help the leather to withstand wearing conditions and the leather should be incorporated with the largest possible quantity of it. Analyses of more and less affected parts of worn damaged heavy boots made from curried leathers showed that during wear the acid value of the grease decreased, much fat had been lost, the water-soluble matter diminished, the p_H value of the latter increased, its colour darkened, and the insoluble mineral matter increased. These results show that products possessing potentially high acid values, *e.g.*, true oils and fats, should not be used in currying, owing to their liability to hydrolyse. Their omission from currying mixtures is impracticable owing to their high affinity for the leather fibres. Further tests on original and worn, curried, heavy boot upper leathers showed that the saponification value of the fat content in worn leathers is less than in the original, and the percentage of unsaponifiable matter is greater in the worn leather. An American split of 16% fat content (85% unsaponifiable matter) was the only leather which was undamaged and satisfactory after wear. Two typical stuffing mixtures, *A* and *B* (*A* containing 40% of cod oil, 30% of "superior" hard grease, and 30% of "inferior" hard grease, and *B* consisting of 3.6% of cod oil, 32.2% of hard grease, 32.2% of "stearine," 28.5% of paraffin wax, and 3.5% of mineral oil), gave the following acid value, saponification value, and unsaponifiable matter, respectively:—(*A*) 22.0, 119.5, 38.4%; (*B*) 40.3, 51.1, 61.25%. Leathers curried with *A* were blackened by iron during wear. The acid value of the "stearine" used in currying is sometimes high (88). Experiments showed that leather cannot be impregnated with rubber, but by mixing a solution of gutta-percha in carbon tetrachloride with oils and fats, and allowing the solvent to evaporate, a product is obtained which can be stuffed into leather, imparting more body and waterproofness. In stuffing leather, all fatty materials of high acid values should be excluded, a minimum amount of true oils and fats and as much unsaponifiable matter as possible should be included. An ideal currying grease would contain about 85% of unsaponifiable matter obtained by the use of mineral greases, wool fat, petroleum jelly, paraffin wax, or beeswax. Tallow or hydrogenated whale oil is of use as a softener and to increase the affinity for the leather. Castor, cod, and linseed oils may also be used for the purpose, but are inferior.

D. WOODROFFE.

PATENT.

Dyeing of furs (B.P. 279,942).—See VI.

XVI.—AGRICULTURE.

Composition of natural organic materials and their decomposition in soil. II. Influence of age of plant on rapidity and nature of its decomposition. S. A. WAKSMAN and F. G. TENNEY (Soil Sci., 1927,

24, 317—333; cf. B., 1927, 951).—The composition of rye plants at different stages of growth and their decomposition in sand and in soil by micro-organisms have been investigated. Young plants contain a much higher percentage of nitrogen than more mature plants, and decompose more rapidly, decomposition being accompanied by liberation of nitrogen in an available form. When the nitrogen content of the plant is about 1.7% it is just sufficient to cover the requirements of the micro-organisms which are active in the decomposition during the first four weeks. If there is more than 1.7% N, the excess is rapidly liberated in an available form, whereas if there is less, an additional source of nitrogen will be required for complete decomposition of the material. Soluble substances are the first to be decomposed, followed by pentosans and cellulose. Plant proteins are readily decomposed and microbial proteins synthesised in their place. Lignins are most resistant and tend to accumulate. C. T. GIMINGHAM.

Effect of tree products on bacteriological activities in soil. II. Forest soils. W. M. GIBBS and H. W. BATCHELOR (Soil Sci., 1927, 24, 351—363; cf. B., 1922, 511 A).—*Azotobacter* occurred in about 25% of the forest soils examined. In most of the samples inoculated with *azotobacter*, the organism remained present for at least 18 months, but only five out of twenty-four soils tested fixed definite amounts of nitrogen when inoculated and supplied with mannitol. Leaves and pine needles exerted a retarding influence on fixation of nitrogen by *azotobacter* in solution; sawdust had little effect. All the soils tested showed ability to accumulate ammonia, but nitrification was poor. Addition of calcium carbonate improved nitrification in 50% of the soils. C. T. GIMINGHAM.

Biological measurement of available soil potassium. D. E. HALEY and F. J. HOLBEN (Soil Sci., 1927, 24, 345—350).—In a preliminary experiment, a close correlation was found between the amounts of potassium in the aerial parts of buck-wheat plants grown to maturity in sand to which known weights of soils were added and the amounts of potassium extractable from the media by 0.2N-hydrochloric acid. C. T. GIMINGHAM.

Effect of hydrogen-ion concentration on the absorption of calcium by a colloidal clay. R. BRADFELD and E. W. COWAN (Soil Sci., 1927, 24, 365—372; cf. B., 1924, 721).—The interactions between calcium hydroxide and a colloidal clay at p_H values between 2.0 and 12.0 have been investigated by analyses of the colloid-free supernatant liquids. In the acid region more bases are liberated than absorbed, whereas the reverse is the case in the alkaline region. The absorption curve is steepest at p_H values above 10.0. There was no evidence of saturation with calcium hydroxide at the highest p_H value obtainable with a saturated solution. Measured at the same p_H value, the amount of calcium absorbed by the clay appeared to be independent of the concentration of calcium added. Potentiometric titration with sodium or barium hydroxide of samples of colloidal clay freed from exchangeable bases by electrodialysis gave absorption curves made up of two rather distinct portions. In the first portion,

covering the acid range, the exchangeable hydrogen was neutralised; in the second, covering the alkaline range, a decomposition of the exchange complex with the formation of simpler silicates and aluminates took place. C. T. GIMINGHAM.

Measurement of physical characteristics of soils. L. H. STAUFFER (Soil Sci., 1927, 24, 373—379).—The cohesion of soils in the wet state may be measured by forcing the soil through a glass tube of known diameter held in a vertical position, and weighing a number of the segments of the soil column which fall from the end of the tube. The average weight of the segments divided by the cross-sectional area of the tube gives the mean force which, acting across a cross-section of 1 sq. cm., is just sufficient to overcome the internal cohesive forces in the soil. The curve relating clay content and cohesion is almost a straight line. An indirect method of measuring the cohesive properties of dry soil conglomerates by means of the modulus of rupture is also described. Cylinders of moist soil are allowed to stand on end until dry, and are then tested as simple beams with a concentrated load at mid-span. The relationship between clay content and modulus of rupture is linear. Shrinkage may be measured on the same cylinders of soil. C. T. GIMINGHAM.

Factors influencing the composition of cabbage and their relation to the quality of sauerkraut. W. H. PETERSON, H. B. PARMELE, and E. B. FRED (Soil Sci., 1927, 24, 299—307; cf. B., 1926, 252).—Late cabbage makes better quality sauerkraut than early cabbage. Storage of the cabbage at low temperatures or slight freezing has little or no effect on the quality, but storage at high temperatures or severe freezing results in a sauerkraut of poor quality. The inorganic constituents, the nitrogen compounds, and the carbohydrates of cabbage are influenced by the type of soil on which it is grown. Detailed figures are given showing the effect of various conditions on the composition of cabbage. C. T. GIMINGHAM.

Decomposition studies of lucerne and sweet clover roots and straw. T. L. MARTIN (Soil Sci., 1927, 24, 309—316).—Comparative experiments on the rates of decay in soil of lucerne roots (1.6% N), sweet clover roots (0.87% N), and straw (0.42% N) indicate that the lucerne decays most rapidly and the straw least rapidly. The rates of decay were measured by determinations of the amount of carbon dioxide evolved, the accumulation of nitrates, formation of humus (by Waksman's method, cf. B., 1926, 958), and by counts of the fungi present at weekly intervals. C. T. GIMINGHAM.

Behaviour of arsenic acid in regard to the absorbing power of soil. C. ANTONIANI (Atti R. Accad. Lincei, 1927, [vi], 6, 107—112).—As regards its absorption by soil, arsenic acid behaves similarly to phosphoric acid, but is absorbed to a less extent. The anion PO_4 is displaced by AsO_4 in solutions of arsenic acid, but not in those of arsenates. T. H. POPE.

Deflocculation and plasmolysis of the coating on soil particles. J. DUMONT and B. GANOSSIS

(Compt. rend., 1927, 185, 1300—1302).—Specimens of a sandy soil, previously dried and sifted, were placed in glass containers to a depth of 16 cm., and the velocity with which distilled water and various 0.1*N*-saline solutions drained through the soil was measured, thus showing the amount of flocculation or deflocculation in the soil caused by the action of the liquids on the limono-colloidal coating of the sandy particles. Distilled water gave constant drainage figures after 48 hrs.; ammonium and sodium carbonate solutions caused deplasmolysis, all drainage ceasing after a few days; potassium carbonate at first leached out much of the humic colloids, the drainage almost ceasing on the third day, but thereafter a steady increase towards the initial value occurred. Calcium chloride solution was applied to the soil rendered impermeable by sodium chloride and soon restored its permeability.

B. W. ANDERSON.

PATENTS.

Diminishing or preventing loss of carbohydrates in root-crops when stored. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 266,695, 9.2.27. Ger., 26.2.26).—The comminuted material is treated with a narcotic such as chloroform, ethylene bromide, toluene, ethylene, carbon dioxide, or generator gas, with which such substances as hydrocyanic acid, sulphur dioxide, or ammonia may be mixed. *E.g.*, sliced chicory roots treated in an airtight container with 0.1% of their weight of chloroform retain their content of inulin for months.

C. T. GIMINGHAM.

Manufacture of mixed fertilisers. I. G. FARBEIND. A.-G. (B.P. 256,972 and 280,447, 11.8.26. Ger., [A] 14.8.25).—(A) A hot concentrated solution of ammonium nitrate is mixed with diammonium phosphate and, if desired, with a potassium or calcium salt, and the mixture solidified by cooling or spraying. (B) The hot solution of ammonium nitrate is mixed with a potassium or calcium salt and, after solidification, diammonium phosphate is added, either in the solid form or as a hot concentrated solution.

C. T. GIMINGHAM.

XVII.—SUGARS; STARCHES; GUMS.

Undetermined chemical losses of sugar in the refining process. I—VI. M. I. NAKHMANOVICH (Nauch. Zapiski [Russia], 1927, 4, 267—274, 291—298, 323—334, 339—354, 358—366, 396—416).—If high temperatures are employed for only 6—8 min. in clarification, only 0.02—0.03% of sugar is lost. When liquors of 99.3—99.6 purity are boiled in a vacuum with steam at 3—4 atm., the reducing effect and coloration are increased: boiling of slightly acid (0.4 c.c. *N* per litre) high-purity liquors containing up to 0.05—0.06% of invert sugar for 60—75 min. causes negligible inversion. Rise of temperature in passing the fillmass from the pan causes increase in the reducing substances. For 0.07—0.4% (dry) of invert sugar, the total chemical loss of white sugar in boiling is 0.04—0.08%. Less pure liquors lose more sugar. Increase in coloration develops at the heating surfaces. In the moulds, for a slightly acid fillmass of reducing effect 0.24—0.28% of invert sugar, passed from the apparatus at 101—102.5°, the accumulation of invert sugar comprised 0.07% of the dry matter, or 0.09% of the dissolved granulated sugar.

For a fillmass containing 0.48—0.80% of invert sugar, the increase in reducing substances was 0.1% (dry) or 0.13% (dissolved sugar). On the whitening tables the invert sugar in the fillmass increased by 15%. Boiling of syrups increased the invert sugar content by 20%.

CHEMICAL ABSTRACTS.

Examination of the early stages of the hydrolysis of starch by dilute acids. R. G. FARGHER and M. E. PROBERT (J. Text. Inst., 1927, 18, 559—571 r).—The rates of hydrolysis of a commercial Japanese farina and of high-grade potato, wheat, maize, and rice starches in the presence of various concentrations of acid, and at temperatures of 40° and above, were followed by means of a modification of Braidy's method for determining copper numbers. For any given conditions of treatment there is an initial stage during which the starch undergoes conversion into soluble starch. During this period the rate of formation of reducing substances gradually increases, subsequently proceeding at a constant rate, dependent on the temperature and the concentration of acid used, in accordance with the relation $N_{Cu} = k(T - t)$, where N_{Cu} is the copper number of the product, T the time of reaction, and t the time required for the complete conversion of the starch into its soluble modification. The latter factor (t) diminishes with rising temperature, is practically eliminated by any pre-treatment of the starch which causes liquefaction, and is ascribed, therefore, to the reactions accompanying the solubilisation of the starch. Measurements using hydrochloric and sulphuric acids of various concentrations at 40°, 50°, and 60° show that the rate of reaction is directly proportional to the concentration of the acid, whilst, as in the case of cotton, the two acids have approximately the same effect in equimolar concentration. Measurements of the time required for complete liquefaction, of the rate at which copper-reducing substances are formed subsequently, and of the speed of dissolution of the hemicelluloses, using 0.2*N*-sulphuric acid, show that for a rise in temperature of 10° the rate of attack of starch is increased approximately four-fold, and as the corresponding figure for cotton is 2.3, the rate of attack of starch with rising temperature increases much more rapidly than that of cellulose. Within limits, therefore, more efficient removal of starch is anticipated at higher temperatures and for shorter periods of treatment than are normally used in the grey-souring of cotton. Potato, maize, wheat, and rice starches differ significantly (but not greatly) in their behaviour towards 0.2*N*-sulphuric acid at 50°, the differences gradually disappearing as the temperature is raised, until at 80° the rates of hydrolysis of rice and potato starches, which show the maximum difference at 50°, become practically identical. The velocity coefficients obtained do not place these starches in the order of their hemicellulose contents.

B. P. RIDGE.

Sugar content of melons. LUTOCHIN.—See XIX.

Determination of aldose sugars. HINTON and MACARA.—See XIX.

PATENTS.

Separation of impurities from molasses and sugar juices by electrolysis. J. DE BRIEY (B.P. 280,321, 13.9.26).—Molasses or sugar juices, diluted or

undiluted, are passed through an inner series of compartments separated by vegetable parchment membrane partitions from an outer series through which purified water is flowing. In the first phase the liquid to be treated is connected with the cathode and the water with the anode, whereby the acids are eliminated and the alkalinity is increased. In the second phase the liquid is brought into contact with the anode and the water with the cathode in order to remove alkalis. Finally the liquid passes continuously between compartments of water constituting cathode and anode for the simultaneous elimination of both acids and salts. Formation of emulsions is prevented by blowing air, steam, or gas through the liquid. F. R. ENNOS.

Preparation from starch of esters of higher fatty acids, soluble in hydrocarbons of the aromatic series. P. BERTHON, ASST. to SOC. DE STEARINERIE ET SAVONNERIE DE LYON (U.S.P. 1,651,366, 6.12.27. Appl., 6.3.23. Fr., 21.12.23).—See B.P. 208,685; B., 1924, 191.

XVIII.—FERMENTATION INDUSTRIES.

Importance in brewing of the ethereal oils from different types of hops. H. LÜERS (Woch. Brau., 1927, 44, 588—590).—The ethereal oils obtained by steam distillation from different varieties of hops vary markedly in quality and amount. When the extraction is effected at ordinary pressure, Californian hops give the lowest yield of raw oil. Slightly more is obtained from Kent hops, whilst those from Saaz and Hallertau contain the greatest quantities. On fractionating the raw oils, the low-b.p. fraction from the Californian hop oil exceeds that from the other oils, whilst the Saaz and Hallertau hop oils contain the less volatile constituents in greatest amount. The high- and low-b.p. fractions from the Kent hop oil are intermediate in amount. When extraction is effected at 2 atm. pressure, the colour of the raw oils is no longer yellow, but green. The yield of oil from the Californian hops is increased, whilst the quantities obtained from Saaz and Hallertau hops are greatly diminished. There is no difference in the aroma of the beers brewed from the same malt and the different varieties of hops, and since the variations in the nature of the oils are not repeated in the beer, it follows that the oils must have been almost wholly volatilised during the boiling of the wort in the copper. The flavours of the resulting beers differ slightly in the amount of bitterness. Saaz hops give an excellent flavoured beer, Californian hops a moderately bitter flavour, and Hallertau hops an excessive bitterness. These differences may be partly due to secondary derivatives produced by the chemical changes undergone by the hop oils during the boiling of the wort. C. RANKEN.

Nitrogenous organic and ammoniacal nutrients in the aeration process of yeast manufacture. H. CLAASSEN (Chem.-Ztg., 1927, 51, 942—943).—The statement by Wohl that the substitution of organic nitrogen by ammoniacal nitrogen as nutrient in the aeration process of yeast manufacture can be made without prejudicing the yield and quality of the yeast, holds good only for nutrient solutions where the amount of nitrogenous pabulum is no more than sufficient for

the needs of the yeast. In brewing practice, where the fermentation liquid contains from 3 to 4 times the optimal quantity of nitrogen, the replacement of organic by inorganic nitrogen is followed by less reproduction and a lower quality of yeast. The added ammonium salts hinder the assimilation of the organic nitrogen, and much of that valuable nutrient is wasted. In nutrient solutions, with a surplus of organic nitrogen and no ammoniacal nitrogen, greater reproduction takes place and the nitrogen content of the yeast is lower. By replacing the organic by inorganic nitrogen, the reproduction is progressively diminished and the nitrogen content of the yeast gradually increases. When 50% of the organic nitrogen is replaced, the reproduction is 14% lower and the nitrogen content of the yeast shows an approximate increase of 2%. C. RANKEN.

Errors in malt analysis due to the want of standardisation of the degree of fineness of the malt meal. E. F. ROTHENBACH (Woch. Brau., 1927, 44, 601).—Variations between duplicate determinations of the extract of a malt may be due to differences in the degree of fineness to which the malt has been ground. The sieves which are used to adjust the Seck grist mill and the fine-meal mill are not standardised, and may deliver with a standard malt varying quantities of malt flour into the three finest fractions. With a sieve made to the specification of the Pfungstadt Brewery, 39.7% of the grist from the Seck mill passed through the three finest sieves in the form of malt flour, whereas with a second set of sieves the same grist yielded 45.2% of flour. With the grist from the fine-meal mill the same two sets of sieves gave 90.1% and 94.6% of fine flour. As the Seck and fine-meal mills are adjusted by means of the sieves to yield on grinding a standard malt, grists of which, 40% and 90%, respectively, pass into the three finest fractions, variations in the screening by the sieves will be repeated in the setting of the mills, with the resulting introduction of errors. C. RANKEN.

Use of luminescent phenomena in the examination of grape and fruit wines. F. M. LITERSCHIED (Z. Unters. Lebensm., 1927, 54, 294—296).—The coloured fluorescence observed in fruit wines illuminated by the filtered light of a quartz lamp can be used as a basis for the examination of wines. The extent to which luminescence can be prevented by previous shaking of the wine with animal charcoal, coupled with the power of amyl alcohol to extract the colouring matter of wine, would appear to render possible the classification of wines as to nature and origin. A. G. POLLARD.

Deterioration of brandy during storage. S. GOY and A. KOEHLER (Z. Unters. Lebensm., 1927, 54, 270—275).—Loss of alcohol from spirit stored in tightly bunged oak casks is small, in ash casks it is considerable, whilst in casks with ill-fitting or open bungs it is very rapid. The importance of testing spirit immediately before selling to users is emphasised. Differences between alcohol determinations made by direct density measurements and by the distillation method are negligible. A. G. POLLARD.

Correction of the hardness of brewing water by lactic acid. H. LÜERS (Woch. Brau., 1927, 44, 385—387).—The neutralisation by lactic acid of a brewing

water containing soluble carbonates improves the quality of the beer brewed from it. The actual and potential acidities of the mash and wort are increased to a degree more favourable for enzymic action, as is shown by the augmented amount of maltose in the wort. The total and "formol" nitrogen in the wort are also higher, but the phosphorus content is lower. On boiling and hopping in the copper, the flocculation is superior and the extract slightly greater. The finished beer brewed from the water neutralised by lactic acid, compared with that manufactured from the water subjected merely to preheating, is paler in colour, more brilliant, has a milder flavour with a non-lasting hop bitterness, a greater capacity for foam production, and an improved head retention.

C. RANKEN.

Alcohol. CARRIÈRE, also SCHLUMBERGER.—See V.

XIX.—FOODS.

Determination of aldose sugars by means of chloramine-T, with special reference to the analysis of milk products. C. L. HINTON and T. MACARA (Analyst, 1927, 52, 668—688).—The quantitative oxidation of aldose sugars may be satisfactorily brought about by means of chloramine-T in conjunction with potassium iodide, but oxidation requires about $1\frac{1}{2}$ hrs. at ordinary temperature. Addition of small amounts of sodium hydroxide had no effect on the oxidations; chloramine-T alone had no oxidising action on dextrose or lactose; sucrose was oxidised less, and lævulose more, than by alkaline iodine under conditions giving full oxidation of the aldose sugar. In the case of clarified milk serum, if this was made slightly alkaline no action of chloramine-T on oxidisable substances occurred other than the slight one on lævulose; complete oxidation of lactose took place with an error not exceeding 0.4% of the total lactose. Lactose in milk serum was determined by washing 25 c.c. of fresh milk into a flask with about 100 c.c. of water and adding 10 c.c. of phosphotungstic acid reagent. After making up to 200 c.c., shaking, allowing to settle, and filtering, 25 c.c. portions of serum are neutralised with 0.1N-sodium hydroxide, a further 3 c.c. of alkali are added, followed by 20 c.c. of 10% potassium iodide and 50 c.c. of 0.05N-chloramine. After being kept at 17—18° for $1\frac{1}{2}$ hrs., 10 c.c. of 2N-hydrochloric acid are added to each flask and the liberated iodine is titrated with thiosulphate solution. A blank experiment is also made and the difference between the titrations calculated to grams of iodine; this value divided by 0.705 gives the amount of lactose in 25 c.c. of serum. To determine sucrose, two chloramine oxidations are necessary, the first as above for lactose. For inversion, 25 c.c. of the original serum, 15 c.c. of water, and 5 c.c. of 6.34N-hydrochloric acid are heated at 60° for 12 min., cooled, and 5 c.c. of 6.34N-sodium hydroxide are added; the solution is neutralised to litmus by 0.5N-sodium hydroxide, cooled, and made up to 100 c.c. 25 c.c. portions are then treated as before with 3 c.c. of 0.1N-sodium hydroxide, iodide, and chloramine, and finally titrated with thiosulphate solution. The difference from the blank titration is calculated as grams of iodine equivalent to the chloramine consumed by the lactose and the inverted

sucrose. Sucrose factors corresponding to varying amounts of chloramine consumed before and after inversion are tabulated, and results obtained by the chloramine and polarimetric methods are compared.

D. G. HEWER.

Importance of the specific electrical conductivity of milk and a new method for its determination. V. GERBER (Z. Unters. Lebensm., 1927, 54, 257—270).—Work previously described (B., 1926, 845) is extended to include the examination of milks of different origin and treatment. From a large number of individual analyses it is concluded that the limiting values for the specific conductivity are $49.0\text{--}51.0 \times 10^{-4}$. Alterations and improvements in the original method are described.

A. G. POLLARD.

Composition and analysis of Danish butter. A. C. ANDERSON (World's Butter Rev., 1927, 1, No. 4, 21—22).—In preparing samples for analysis, the stirring method is more accurate than the shaking method. Salted Danish butter contains about 0.9% and unsalted about 0.08% of salt.

CHEMICAL ABSTRACTS.

Green colour in butter. A. A. RAMSAY, A. M. BROWN, and H. H. RANDELL (Agric. Gaz., N.S. Wales, 1927, 38, 475—480).—The development of green colour in butter was ascribed to decomposition of protein with formation of melanins. It appeared only when the herbage was infested with aphids.

CHEMICAL ABSTRACTS.

Vitamin content of technical soya-phosphatide preparations used in margarine factories. A. SCHEUNERT (Z. Unters. Lebensm., 1927, 54, 302—307).—Soya-phosphatide preparations contain only traces of vitamin-A and no antirachitic vitamin. By treatment with ultra-violet light active antirachitic properties can be developed.

A. G. POLLARD.

Composition and examination of milk pastry. A. GOTTFRIED (Z. Unters. Lebensm., 1927, 54, 279—281).—The use of whole milk or diluted milk in pastry mixing is detected by a determination of the alkalinity, lime, and phosphate in the ash. From a number of analyses the mean values for alkalinity (number of c.c. of N-NaOH per 20 g. of air-dry substance), calcium oxide (g.), and phosphate (g. of PO_4) were:—(a) for whole milk, 2.6, 0.101, 0.45; (b) for diluted milk, 1.2, 0.068, 0.43; and (c) without milk, 0.1, 0.048, 0.36.

A. G. POLLARD.

Panification with soya flour. I. D. COSTA (Annali Chim. Appl., 1927, 17, 524—530).—The percentage composition of wholemeal flour from the seeds of yellow *Soja hispida* and that of the flour obtained from seeds extracted with light petroleum (in brackets) were found to be: moisture 10.40 (10.80); protein ($\text{N} \times 6$) 37.98 (47.34); cellulose 5.05 (5.35); fat 18.32 (0.37); non-nitrogenous extractives 23.83 (30.16); ash 4.42 (5.98). Loaves were prepared from mixtures of 75% wheaten flour or the 82% flour now compulsory in Italy with one fourth or one ninth of its weight of soya flour from which the unpleasant flavouring materials were first removed. The bread proved heavy and unappetising, and, even with the smaller proportion of soya flour, not readily tolerated in cases of weak digestive powers.

T. H. POPE.

Wood's light for the detection in wheat flour of extraneous and harmful seeds and of extraneous mineral substances. G. CAPELLI (*Annali Chim. Appl.*, 1927, 17, 513—519).—When wheaten flour containing 2% or more of pea meal is exposed to Wood's light, the particles of pea meal appear as bright orange-flesh-coloured spots against the violet-blue nacreous mass of the wheaten flour. When more than 10% of the pea flour is present, the orange-flesh-coloured fluorescence begins to predominate. This procedure fails to reveal marble, talc, silica, alumina, soil, gypsum, magnesia, copper sulphate, zinc sulphate, barium sulphate, or kaolin in wheaten flour, but in the 82—84% flour now obligatory in Italy it allows of the detection of 0.3% or more of zinc oxide. T. H. POPE.

Loss of mineral substances from potatoes during ordinary culinary preparation. R. BERG (*Z. Unters. Lebensm.*, 1927, 54, 314).—Losses of mineral matter during the boiling of peeled potatoes depend largely on the "mealy" or other consistency of the potato. Such losses are partly attributable to direct extraction of soluble material and partly to physical disintegration of the flesh of the potato, and may be sufficient seriously to affect the character of the potato as a base-carrying foodstuff (cf. Griebel and Miermeister, B., 1927, 236).

A. G. POLLARD.

Sugar content of water- and other melons. S. N. LUTOCHIN (*Z. Unters. Lebensm.*, 1927, 54, 281—289).—The total sugar content of melons varies considerably with the variety, and also among individual samples of the same variety. In most cases *laevulose* is present in the greatest proportion, followed in order by dextrose and sucrose, the mean values being respectively 52%, 33%, and 14% of the total sugars. The total yield of sugar per melon varies enormously. *Laevulose* and sucrose seem to be stored up principally during the ripening period. The nature of the soil affects the sugar content of melons growing therein. A. G. POLLARD.

Importance of water-melon syrups as foods. S. LUTOCHIN (*Z. Unters. Lebensm.*, 1927, 54, 290—291).—Analyses of water-melon syrups are recorded.

A. G. POLLARD.

Changes in the flesh and in the brine of salted fish. G. DRUCKER (*Z. Unters. Lebensm.*, 1927, 54, 253—257).—Various fish were pickled in common varieties of pickling salt. Variations in the amounts of calcium and magnesium in the salt produced only slight alterations in the taste of the fish. The quality of the product was mainly affected by the freshness of the original fish, the method of preparation, and the temperature and concentration of the brine used. The degradation of protein matter in the softer flesh of fish during pickling may proceed to a considerable extent without detracting from their good quality and without any rancidity being apparent. After pickling for 255 days, the concentration of nitrogen titratable with "formol" in the tissue fluids represented 81% of the total nitrogen. A. G. POLLARD.

Bottled cocoa beverages. J. B. WILSON and W. R. TURNER [with J. W. SALE] (*Amer. Food J.*, 1927, 22, 244—246).—An investigation of the persistence of hydrogen peroxide in bottled cocoa beverages; the

effect of the presence of yeast and bacteria is also examined. Hydrogen peroxide was present in all but one of the cocoa beverages. The potassium iodide and benzidine methods for the determination of hydrogen peroxide are preferred. CHEMICAL ABSTRACTS.

Metals for cooking utensils. SPRINKMEYER.—See X.

Bromine-iodine values of fats. VAUBEL. **Butter fat and coconut oil.** KLOSTERMANN and QUAST.—See XII.

PATENTS.

Heat-treatment of cheese. A. E. WHITE. From LAKESHIRE CHEESE CO. (B.P. 280,257, 5.7.26).—Finely-divided cheese is passed continuously through a treating vessel, where it is subjected momentarily to the direct action of steam which sterilises it and reduces it to a semi-liquid or plastic condition, but does not destroy its characteristic flavour. While still fluid it is impregnated with carbon dioxide gas, packed in the warm state, and allowed to cool. [Stat. ref. to B.P. 270,046.]

F. R. ENNOS.

Manufacture of fodder from sugar cane. A. E. KIENZLE (B.P. 280,284, 11.8.26).—After removal of the leaves, the sugar cane is cut into small pieces, dried in the sun or by artificial means, and ground to a fine powder while being heated at a temperature insufficient to caramelize the sugar. The milled product is graded according to fineness, and the medium quality, forming about 75—80% of the whole, is mixed with ground maize cobs etc. and used as a mixed fodder.

F. R. ENNOS.

Manufacture of substances from sugar cane for use in the preparation of food and beverages. A. E. KIENZLE (B.P. 280,432, 11.8.26).—Sugar cane is cut into chips or discs etc., dried, and roasted until the sugar is more or less caramelised. The material is powdered and the coarse fibrous ingredients are removed by sifting, either before or after roasting, and the product is used as a colouring and flavouring ingredient for foods and beverages or as a coffee substitute.

F. R. ENNOS.

Alimentary industrial product consisting of the juices of oranges or lemons, with their pulps and free from their essences. A. T. MEZQUITA (B.P. 256,985, 12.8.26. Spain, 12.8.25).—The ripe, sound fruit is grated in order that the essence may be subsequently extracted from the separated rind, cut in halves, and squeezed to liberate the juice, pulp, seeds, and refuse. The mass is then strained first to remove the seeds and refuse, and again to separate the pulp from the juice, and the latter is subjected to a low temperature (about -10°) to freeze out the water, the frozen particles of which are separated centrifugally. The pulp is then added to the concentrated juice, and bottled with addition of a small quantity of a suitable preservative. Each bottle of juice and pulp is accompanied by another containing sufficient essence to impart the natural aroma thereto at the time of mixing. F. R. ENNOS.

Manufacture of aerated waters, sparkling drinks, etc. BRIT. DYESTUFFS CORP., LTD., C. HOLLINS, and E. CHAPMAN (B.P. 280,302, 19.8.26).—Wetting-out or dispersing agents, viz., sulphonic acids of high mol. wt., particularly the *isopropylated* sulphonated mineral

oil fraction of B.P. 274,611 (B., 1927, 841), are added to aerated waters, flavouring syrups for soda-fountains, effervescent salines, etc. The treated substances give a more persistent froth, and the liquid retains its "sharpness" for a longer time.

C. HOLLINS.

Manufacture of bread. H. A. KOHMANN, Assr. to FLEISCHMANN Co. (U.S.P. 1,651,717—8, 6.12.27. Appl., 29.6.26).—See B.P. 244,489; B., 1927, 376.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Ephedrine assay of Chinese *Ephedra*. C. T. FENG and B. E. READ (J. Amer. Pharm. Assoc., 1927, 16, 1034—1039).—A critical examination of the methods of ephedrine assay of Chinese Ma Huang has been made. The following methods of extraction, followed by treatment of the extract with alkali and chloroform, were used. The figures represent the amounts of total alkaloid calculated as ephedrine, and pure ephedrine (by biuret assay) respectively: hot acetic acid, 0.993%, 0.614%; hot hydrochloric acid, 1.32%. —; cold aqueous potassium carbonate, 1.12%. —; cold acetic acid, 0.302%. —; cold sulphuric acid, 0.283%. —; adsorption by fuller's earth from a water extract of the material extracted with hot acidified alcohol, 0.961%, 0.512%. The ammonia-chloroform method has been studied, and it is shown that, for complete liberation of the alkaloids, the addition of a very large excess of ammonia is necessary. A physiological method for confirming the chemical methods is outlined.

E. H. SHARPLES.

Quantity of alkaloid in *Hyoscyamus* and a new method for its evaluation. H. R. WATKINS and S. PALKIN (J. Amer. Pharm. Assoc., 1927, 16, 1039—1044).—Total alkaloid in *Hyoscyamus niger* has been determined by soaking the powdered drug in a mixture of strong ammonia solution, alcohol, and ether for several hours, and then extracting with ether in a continuous extraction apparatus. After purification of the extract the alkaloid is extracted from aqueous solution by means of another automatic extractor (cf. B., 1925, 566) and determined by titration. This method yields from two to three times as much alkaloid as that obtained by the U.S.P. IX and X methods, and the alkaloid by physiological test shows the full hyoscyamine mydriatic power (cf. B., 1927, 399).

E. H. SHARPLES.

Constituent of commercial ethyl ether. H. KING (Nature, 1927, 120, 843).—Commercial samples of ethyl ether, whether exposed to light or not, contained ethyl vinyl ether.

A. A. ELDRIDGE.

Sterilisation of hexamethylenetetramine solutions. U. CAZZANI (Boll. Chim. farm., 1927, 66, 674—676).—Hexamethylenetetramine solutions to be used for endovenous injection should be freshly prepared with cold water, and should be either sterilised by heating at 60—70° or passing through a filter-candle, or prepared aseptically. At 120°, or even 100°, appreciable decomposition of the amine occurs.

T. H. POPE.

Solubilities of drugs in glycerin. J. A. ROBORGH (Pharm. Weekblad, 1927, 64, 1205—1209).—Solubilities are recorded of some twenty medicinal compounds in glycerin of d 1.23 and 1.26, determinations being made at 20° both by shaking with excess of solute and by cooling a solution saturated at 80—90°. Supersatura-

tion is very general. Where water of crystallisation is present, the solute may be dehydrated by the solvent. Phenol, tannin, sodium arsenate, and sodium pyrophosphate had the highest solubilities of the substances examined, and theobromine, guaiacol carbonate, phenacetin, zinc valerate, and stearic acid the lowest.

S. I. LEVY.

Determination of aldehyde in essential oils, with particular reference to the determination of citronellal in Java citronella oil and citral in lemon oil. C. T. BENNETT and M. S. SALAMON (Analyst, 1927, 52, 693—695).—To 5 g. of hydroxylamine hydrochloride dissolved in 9 c.c. of hot water are added 80 c.c. of 90% alcohol (d 0.833) and 2 c.c. of Bromophenol Blue solution (1 g. with 3 c.c. of 0.05*N*-sodium hydroxide, and made up to 25 c.c. with water), and the whole is neutralised, if necessary, with 0.5*N*-alcoholic alkali, and made up to 100 c.c. with more 90% alcohol (rectified, not industrial, spirit should be used). 20 c.c. of the hydroxylamine hydrochloride are added to 2 g. of citronella oil, and the alcoholic alkali is run in until the sharp end-point is reached. The values thus obtained are lower than those by the Dupont and Labaune method, but are regarded as being more correct. The method was successfully applied to the determination of citral in lemon oil and to several other oils containing aldehyde.

D. G. HEWER.

Production of essential oils from Irish-grown plants. II. Cultivation of *Mentha piperita*, and further experiments on the winning of lavender oil. J. REILLY and C. BOYLE (Econ. Proc. Roy. Dublin Soc., 1927, 2, 285—291).—The varieties and cultivation of *M. piperita* are discussed with a view to the economic production of oil of peppermint in Southern Ireland. Lavender oil, from English plants after cultivation for one year in Cork, had d 0.8968, n 1.4679, α $-5^{\circ} 20'$, acid value 0.7, and ester value 14. An oil from ordinary nursery lavender plants which had been growing in Cork for 7 years resembled spike lavender oil, and had d 0.890, n 1.4650, α $-7^{\circ} 30'$, acid value 2.4, and ester value 69 (cf. B., 1926, 462).

E. H. SHARPLES.

Production of essential oils from Irish-grown plants. III. Oil of peppermint. J. REILLY and J. TAYLOR (Econ. Proc. Roy. Dublin Soc., 1927, 2, 292—297).—Seventy pounds of green herb from 300 Mitcham plants grown in Cork during 1926 gave a yield of about 0.2% of peppermint oil having d 0.903, n 1.4631, α -19.5° , menthol, total 66.2%, combined 3.27%, and solubility in 70% alcohol 1 in 2.3. Values for oil from "black" and "white" peppermint and for first and second crop Australian and Sicilian oils are given, and both these and reports from independent experts show that the oil from Irish-grown plants compares favourably with the best varieties of peppermint oil. Analytical figures showing the variations in nine types of peppermint oil are given and different methods of distillation are discussed. The yield of oil at different periods of the plant's growth has been examined. The season of flowering is the period of highest yield, and the largest amount of oil was obtained from leaves and flowering tops, and was of better quality than the oil from the stalks.

E. H. SHARPLES.

Production of essential oils from Irish-grown plants. IV. Oil of camomile. J. REILLY and P. J. DRUMM (Econ. Proc. Roy. Dublin Soc., 1927, 2, 298—301).—Distillation of the hand-picked flowers from a small plot of *Anthemis nobilis* (single variety) grown in Cork gave a blue-coloured oil having d_{20}^{20} 0.919, n_D^{20} 1.4464, α_D^{20} +2° 15', acid value 2.0, and ester value 172. These figures are comparable with those of a camomile oil from Mitcham-grown plants. The cultivation of the plant and the properties of the oil are described.

E. H. SHARPLES.

PATENTS.

Manufacture of colourless melted [pharmaceutical] products. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 272,875, 20.5.27. Ger., 18.6.26. Addn. to B.P. 255,434; B., 1926, 931).—Colourless products are obtained by melting 4-dimethylamino-1-phenyl-2:3-dimethyl-5-pyrazolone with soporifics in an inert atmosphere. Compounds are described in which 1 mol. and $\frac{1}{2}$ mol. of phenylethylbarbituric acid, respectively, (m.p. 130—133° and 100—129°); 1 mol. of trichloroethyl carbamate (m.p. 73—75°) and also $\frac{1}{2}$ mol., and 1 mol. of bromodiethylacetylcarbamide (m.p. 82—86°) are used.

B. FULLMAN.

Preparation of anti-diabetic material from pancreas. J. R. MURLIN (U.S.P. 1,646,553, 25.10.27. Appl., 27.3.24).—Fresh, finely-divided pancreas is heated with 0.2N-hydrochloric acid at 90°, and the liquid is filtered, adjusted to p_H 4.1, and refiltered. The filtrate is saturated with sodium chloride, and the precipitate formed is dissolved in 70% alcohol and reprecipitated by addition of butyl alcohol. It is administered *per os* mixed with citric acid to prevent destruction of the anti-diabetic principle by trypsin.

T. S. WHEELER.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Nature of desensitisation. V. SIHVONEN (Z. wiss. Phot., 1927, 25, 1—9).—On the basis of Baur's theory of photolysis it is shown that desensitisation can be regarded as a special case of sensitisation, the important fact for this view being that desensitisation holds only for the blue light which is absorbed by the silver bromide and not for the wave-lengths absorbed by the dyes. The silver bromide is the light receiver and the dye is the substance acted on. The silver bromide is regarded as the sensitiser for the photolysis of the dyestuff, and gives up its light content to the surroundings; otherwise the light would act on the silver bromide itself, which would be decomposed. In order for these views to be correct, it follows that under the same conditions silver bromide with no desensitiser deposits more silver on exposure than silver bromide with desensitiser, and that for the difference in the amount of silver set free there must be an equivalent amount of dye reduced. To test the views silver bromide emulsions were exposed with anodic depolarisers (organic reducing agents) in presence and absence of dye, and the results of the experiments are given.

W. CLARK.

Photochemical nucleus destruction. LÜPPO-CRAMER (Z. wiss. Phot., 1927, 25, 25—28).—Densograph curves are given showing that in the case of pre-exposed

plates bathed in Phenosafranin and bromide, a bleaching effect is produced on subsequent exposure which, however, reverses at higher intensities giving a normal image again. In absence of bromide the density of image may even rise to a higher value than that of the original image. If the second exposure is made through a yellow filter, bleaching alone results, and no reversal is found. The second reversal in the bleach-out reaction is ascribed to the light penetrating deeply into the grains where the action of the desensitiser is not felt, so that the deeper lying parts of the grains are affected. With Pinakryptol Green and Yellow the second reversal does not occur, but merely a bleaching, either in unfiltered or blue light.

W. CLARK.

Intensification of the latent image. LÜPPO-CRAMER (Z. wiss. Phot., 1927, 25, 23—25; cf. B., 1927, 268).—In 1915 the author made experiments similar to those of Wightman and Quirk (*loc. cit.*), and came to similar conclusions (Phot. Korr., 1915, 135). The water effect observed by Wightman and Quirk has also been observed by the author (B., 1927, 714, 715).

W. CLARK.

PATENTS.

Light-sensitive films. U. DIEM-BERNET (F.P. 617,929, 19.6.26. Switz., 17.5.26).—Instead of gelatin, substances such as acetyl- or citryl-cellulose, cellophane, etc. are used. These materials are resistant to mechanical influences, and can be used without a support, and can therefore be exposed from either side. Several such films may be united together or with colour filters.

W. CLARK.

Manufacture of bodies of photographic desensitising action. I. G. FARBERIND. A.-G. (B.P. 262,816, 13.12.26. Ger., 11.12.25).—Desensitisers made by condensing a methylpyridine or methylquinoline base with an aldehyde of a negative character (cf. G.P. 396,402; B., 1924, 902) are not satisfactory because they are not sufficiently soluble, and their desensitising action is not very high. These faults are remedied by the introduction of certain groups at determined points in the molecular complex. Suitable desensitisers are produced by condensing together an alkyl sulphate of a *p*-alkyloxyquinoline with a *m*-nitrobenzaldehyde. The products have only a faint yellow colour.

W. CLARK.

Photographic emulsions. I. G. FARBERIND. A.-G. (B.P. 271,475, 17.5.27. Ger., 20.5.26).—Gelatins which are unsuitable for making emulsions on account of their fogging tendencies may be rendered useful for making clear, high-speed emulsions if glyoxaline or its derivatives are added to the gelatin or to the emulsion in the course of manufacture. Glyoxaline derivatives produced by the degradation of proteins may be used.

W. CLARK.

Decoration of fabrics by the reducing action of light on silver salts. M. MICHELS (G.P. 441,690, 4.5.26. Addn. to G.P. 441,150; B., 1927, 861).—Instead of a ferrocyanide or metal oxide mordant, a mordant suitable for all basic dyes is formed from complex copper compounds. The basic dyes form with the complex acids mordant dyes which form coloured lakes with cuprous ions. The resulting images are very stable and pure, and have great depth of colour.

W. CLARK.

Colour images, especially for multi-colour photography. D. LIERG, K. POKORNY, and L. ROSS (Austr. P. 105,591, 9.8.24. Cf. B.P. 245,470; B., 1926, 997).—A glass or film base is coated first with a rubber solution and then with gelatin hardened with formaldehyde. On this substratum is coated an emulsion containing a silver halide, tannin, and in some cases a further substance which forms a lake with certain dyes. The negative is printed on this layer from the glass or celluloid side, developed, and treated first with chromic acid solution and then with hot water. A gelatin-coated transfer paper is then rolled on and stripped after drying.

W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

Evolution of gases from heated nitrocellulose powders. J. DESMAROUX (Mém. Poudres, 1927, 22, 245—258).—The nature of the gases which are evolved when nitrocellulose powders are heated at the temperatures employed in stability tests has been investigated. The temperatures employed were 108.5° and 75°. The gases evolved from powders maintained at these temperatures were collected at certain intervals and analysed for carbon monoxide and dioxide, nitrous oxide, and nitric oxide. At 108.5°, the chief constituents were carbon dioxide and nitric oxide; nitrous oxide was absent. During the heating of the powder the amount of nitric oxide evolved increases to a maximum and then decreases. The presence of a stabiliser in the powder increases the hours of heating which are necessary for the evolution of a certain volume of gas. Evidence is adduced showing that the reaction following heating at 75° differs from that at 108.5°.

S. BINNING.

Microscopical examination of B-powders. J. DESMAROUX (Mém. Poudres, 1927, 22, 259—284).—The form in which "C.P. 1" (guncotton) exists in B-powders has been investigated microscopically. Examination of C.P. 1 by itself showed that the structure of the cotton fibre is not altered during nitration, but is destroyed by pulping. "C.P." is shown to be a mechanical mixture of either completely soluble or completely insoluble fibres. By staining with Oxydianiline Black J.W.R. and Lyon Blue B, properly dehydrated powders can be distinguished from those poorly dehydrated, and also washed from unwashed powders. Directions are also given for the differential staining of C.P. 1 and C.P. 2 (soluble nitrocellulose).

S. BINNING.

PATENTS.

Propellant powder. C. H. KECK (U.S.P. 1,651,459, 6.12.27. Appl., 27.7.26).—The powder contains 85—99% of nitrocellulose and 15—1% of nitrated castor oil.

F. G. CROSSE.

Removal of diphenylamine from smokeless powder. D. C. BUTTS, Assr. to HERCULES POWDER CO. (U.S.P. 1,650,689, 29.11.27. Appl., 30.7.26).—The powder is treated with boiling isopropyl alcohol.

H. ROYAL-DAWSON.

XXIII.—SANITATION; WATER PURIFICATION.

Physico-chemical study of scale-formation and boiler-water conditioning. R. E. HALL, G. W.

SMITH, H. A. JACKSON, J. A. ROBB, II. S. KARCH, and E. A. HERTZELL (Min. Met. Invest., Carnegie Inst. Tech. Bur. Mines, Bull., 1927, 24, 239 pp.).—Boiler-water conditioning consists of the maintenance at the evaporating surfaces of ratios between the acidic radicals originally present and introduced, the numerical values of which depend on the characteristics of boiler operation and the chemical used. The behaviour of various added chemicals is discussed, together with conditions of corrosion. The solubilities of calcium sulphate and phosphate are recorded. The decomposition of soda ash is not lessened by the presence of organic matter. The corrosive action of magnesium salts is specific only above a critical concentration. Formulae applicable to control and prediction are given.

CHEMICAL ABSTRACTS.

Sodium aluminate as an adjunct to alum for the coagulation of public water supplies. S. T. POWELL (Amer. J. Pub. Health, 1927, 17, 804—809).—When sodium aluminate is employed as an adjunct to alum, the filtered water contains less free carbon dioxide and alumina. Technical advantages are described.

CHEMICAL ABSTRACTS.

Micro-determination of carbonate carbon [in lake water]. G. KEMMERER and L. T. HALLETT (Ind. Eng. Chem., 1927, 19, 1352—1354).—In the determination of carbon in residues obtained by evaporating lake waters, micro-combustions (A., 1927, 269) are made at 900—950° to decompose all the carbonates, and the carbonate carbon is then determined on 5—15 mg. portions of the residues by decomposition with hydrochloric acid in a specially designed apparatus, phosphorus pentoxide or dehydrite being used as the drying agent and ascarite as the carbon dioxide absorbent. For residues containing up to about 8½% of carbonate carbon, the average error on duplicate determinations was less than 0.1%.

F. R. ENNOS.

Germicidal efficiency of sodium hydroxide, sodium carbonate, and trisodium phosphate at the same hydrogen-ion concentration. M. LEVINE, E. E. PETERSON, and J. H. BUCHANAN (Ind. Eng. Chem., 1927, 19, 1338—1340).—In solutions of the same p_H value sodium carbonate was found to possess a greater germicidal power than trisodium phosphate, which was itself superior in this respect to sodium hydroxide. The times required by solutions of the carbonate, phosphate, and hydroxide, at 70°, and having p_H 11.35, to kill 99.9% of the bacteria the initial count of which was 1 million per 5 c.c. of solution were 120, 163, and over 220 min., respectively. For a given alkali the killing time decreased with increasing p_H value, but this value alone afforded no dependable index of the germicidal efficiencies of different alkalis.

Hardness of brewing water. LÜERS.—See XVIII.

PATENTS.

Preparation of a medium for the treatment of water [germicide]. H. REINBOLD (U.S.P. 1,646,596, 25.10.27. Appl., 10.8.25).—Anhydrous sodium hydroxide is treated with bentonite, and the product is saturated with chlorine.

T. S. WHEELER.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

FEBRUARY 3, 1928.

I.—GENERAL; PLANT; MACHINERY.

Simplified capillary-tube plastometer. J. L. ST. JOHN (Ind. Eng. Chem., 1927, 19, 1348—1349).—A horizontal capillary-tube plastometer is constructed from a short, soft glass test-tube with side tube blown in near the bottom for the pressure lead. The top is closed by a rubber stopper pierced for a long, thick-walled, capillary tube which has been accurately calibrated with mercury. The test-tube is filled with the material to be studied, and the rate of flow, at a given temperature and under a given pressure, along the capillary is measured with a stop-watch. The mobility is then calculated as prescribed by Kelly for a more elaborate apparatus. Examples showing fairly concordant results are given of tests with flour-water suspensions and with reconstituted dry-skim milk.

C. IRWIN.

Air-jet lift [for liquids]. S. C. MARTIN (Ind. Eng. Chem., 1927, 19, 1346—1348).—In an air-lift (emulseur) raising water, the starting pressure $P = 0.434S + F$, where S is the starting submergence and F the transmission in the air line in lb./sq. in. The working air pressure $P' = 0.434(S' - V/2g) + F$, where S' is the working submergence and V the water velocity in ft./sec. The quantity of free air required $= WL/20$ where W is the quantity of water in cub. ft./min. and L the total lift in feet. The overall efficiency from the compressor steam cylinder falls from 50% with a 100 ft. lift to 20% with lifts of 900—1000 ft. An application of the air-lift for elevating sulphuric acid is described. The advantage of the method is in the absence of moving parts and its consequent applicability to gritty or corrosive liquids.

C. IRWIN.

Hydraulic lift for gas-analysis apparatus. E. J. TAUCH (Ind. Eng. Chem., 1927, 19, 1349).—The mercury-levelling bulb of a gas-analysis apparatus is attached to a brass plunger working within a brass cylinder of 2.5 cm. diameter. This is raised or lowered at any desired speed by the rotation of a 3-way cock connected to water under pressure. The transference of gas samples to or from absorption pipettes is thus performed mechanically; particularly accurate results are obtained.

C. IRWIN.

Boiling-point apparatus for calibrating thermocouples. T. DE VRIES (Ind. Eng. Chem., 1927, 19, 1311).—To prevent superheating of vapours used for calibrating thermocouples, the vapours are caused to bubble through some of the liquid before passing around the thermo-junction which was as usual enclosed in a glass tube filled to 1 cm. with paraffin. The inner tube containing the thermocouple heated by the vapours from an outer tube is sufficiently protected without the use of a radiation shield.

C. A. KING.

Protective tubes for thermocouples. FORD and OSBORNE.—See XIX.

PATENTS.

Furnace. H. TIELKEMEYER (U.S.P. 1,650,749, 29.11.27. Appl., 23.7.27).—A vessel to be heated is supported within a cylindrical enclosure with an annular space between them, and the enclosure is mounted within an outer casing. A partition is provided in the enclosure above the vessel, and a pipe connects the annular space with the space above the partition.

H. HOLMES.

Pulverised fuel fired furnaces. CLARKE, CHAPMAN & Co., LTD., and W. A. WOODSON (B.P. 281,023, 27.8.26).—Hot air for drying pulverised fuel during grinding is produced by drawing external air through zig-zag passages constructed in walls upstanding from the bottom of a combustion chamber. The cool walls also aid in preventing formation of slag in the chamber.

B. M. VENABLES.

Gas-heated furnace. J. DELWICHE (U.S.P. 1,650,498, 22.11.27. Appl., 2.2.27).—A heating space is disposed between an outer shell and an inner shell constituting a fire box, and a mound of ashes on the grate prevents up-draught through the grate. The mound is covered by overlapping segments of sheet asbestos leaving a central opening to accommodate a gas pipe provided with a delivery end above the mound.

H. HOLMES.

Regenerative gas-fired furnaces, more particularly of the open-hearth type. W. SCHUCANY (B.P. 281,002, 23.8.26).—A reversing regenerative furnace is provided with water-cooled flaps in the gas and air ports, two in each port at each end, which are used to regulate the proportion of gas to air, the amount of preheating of each, and the velocity and direction of exit into the furnace. The latter are varied continually so that the gas and air streams always intersect on the surface of the bath and the point where they impinge wanders to and fro. The outlet gases are divided up by the flaps at the other end so as to give the desired relative preheating to the gas and air on next reversal. An elaborate control gear is described.

B. M. VENABLES.

Kiln. A. HANSEN (U.S.P. 1,649,029, 15.11.27. Appl., 27.3.26).—The kiln chamber is provided with a hollow floor and with hollow walls arched over the top. Fire holes at one side open each into a corresponding flue formed by partitions within the hollow floor. Each flue is extended vertically up the further side, thence diagonally over the top, vertically down the near side, again through the hollow floor between the portions adjacent to the fire holes, and connected by a longitudinal tunnel to the stack.

H. HOLMES.

Tubular evaporators. G. W. RILEY, and G. SCOTT & SON (LONDON), LTD. (B.P. 280,666, 14.9.26).—The liquor in a multi-tubular evaporator is given forced circulation by means of a centrifugal impeller. In the vertical type the circulation of the liquor in the tubes is up or down the central tubes and down or up the outlying tubes according to whether the centrifugal impeller is above or below the tube bundle. Deflectors may be used to spread out and slow up the current at the bottom reversal, so as to permit solid matter to settle out. In the horizontal type, where the liquor will be outside the tubes, division plates may be used to keep the streams separate. B. M. VENABLES.

Prevention and removal of incrustation in boilers. J. BILLWILLER (B.P. 257,915, 24.8.26, Ger., 1.9.25).—A resin acid, but not the resin itself, is added to the water. Since the acid is difficult to dissolve, it may first be mixed to a paste with glycerin, wood gum, dextrin, or tragacanth and water, or dissolved in alcohol. B. M. VENABLES.

Heat exchanger. W. H. KNISKERN, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,649,120, 15.11.27, Appl., 16.8.23).—The main shell is provided with a top inlet and a bottom outlet header space each between an end plate and a tube sheet, and a nest of tubes connects the sheets. Core rods defining annular passages through the tubes are provided with uniformly spaced centralising projections. Some of these rods are supported upon the lower end plate free from contact with a baffle at the inlet, whilst others have flattened ends for suspension free from contact with a similar baffle at the outlet. H. HOLMES.

Heat-exchange apparatus, including feed-water heaters or economisers. W. ST. JOHN'S-FINDLAY (B.P. 280,266, 5.8.26).—A construction of heat exchanger which gives counter-current flow of the gas and liquid, and in which, in the case of boiler-feed water, the water in the tubes is held at a moderate pressure by the use of pumps before and after the heater. B. M. VENABLES.

Heat-exchange device. G. PAGEL, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,651,900, 6.12.27, Appl., 9.11.26, Ger., 31.12.25).—A heat exchanger of the condenser type is formed in sections, to which the hot fluid is supplied separately. Means are provided for apportioning the cooling fluid to the separate sections and supplying it to them in parallel. H. HOLMES.

Recuperator. W. A. MORTON, Assr. to AMSLER-MORTON Co. (U.S.P. 1,652,210, 13.12.27, Appl., 21.5.26).—The air to be heated traverses passages between which the waste furnace gases are passed. The distribution of the air is such that a greater volume traverses the passages subjected to the higher temperatures. H. HOLMES.

Heat-insulating coverings. R. W. ANDERSON (B.P. 279,913, 27.4.26).—Insulating material of hydrated magnesium carbonate is separated from the article to be insulated by a covering of a strong, non-metallic insulating material, *e.g.*, Portland cement, W. G. CAREY.

Rotary cylinder drying apparatus. I. W. BENSON and T. D. DENHOLM (B.P. 280,740, 18.1.27).—A form of construction of rotary cylinder is claimed in which heated

air is admitted at one end, also through ports left between the overlapping plates of the circumferential joints. B. M. VENABLES.

Disintegrating or mixing apparatus. AFRICAN SELECTION TRUST, LTD., C. W. BOISE, and W. R. DEGENHARDT (B.P. 280,276, 10.8.26).—An apparatus specially suitable for clayey material comprises a rotary drum, provided in the inside with inclined blades some of which propel the material in a forward direction and others send it back again, the throwing effect of the former being greater than that of the latter. The blades in different rows are staggered, and some (or all) are adjustable as to inclination from outside the shell. B. M. VENABLES.

Mixing and disintegrating apparatus. F. M. WICHMAN and A. P. HARTLAPP, Assrs. to U.S. SMELTING, REFINING, & MINING Co. (U.S.P. 1,649,939, 22.11.27, Appl., 7.4.25).—A table and a support above it carrying ploughs and cutting discs are provided with means for producing a relative rotation between them. Material fed on to the table is engaged alternately by the ploughs and the discs, and at the same time is deflected toward the periphery of the table. H. HOLMES.

Grinding mill. R. J. DOREY (B.P. 281,062, 23.10.26).—The grinding is effected between a serrated container and serrated "piston," which rotates, and has also an up-and-down motion. A "piston ring" is provided which effects the final grinding by expanding close to the outer wall. B. M. VENABLES.

Compound mill. P. GOEBELS, Assr. to G. POLYSIUS (U.S.P. 1,650,508, 22.11.27, Appl., 15.10.26, Ger., 17.5.26).—In a pulverising mill a rotary casing is divided into compartments by transverse partitions, and all compartments except the first are subdivided by longitudinal partitions. The latter are relatively angularly displaced in the several compartments. H. HOLMES.

Mill. J. W. BOWMAN (U.S.P. 1,651,319, 29.11.27, Appl., 19.7.26).—A rotary shaft carrying spaced radial arms is mounted in a casing with a foraminous lower portion. The outer end of each arm is provided with transversely-spaced longitudinal slits, and the material between the slits is offset to constitute grinding teeth, one of which is in line with the arm, and others are oppositely and laterally inclined. H. HOLMES.

Pulverising apparatus. RILEY STOKER CORP., Assees. of R. S. RILEY and O. CRAIG (B.P. 260,952, 13.8.26, U.S., 7.11.25).—The pulveriser has two disintegrator compartments, the entrance to the first and outlet from the second are near the axis, but the transfer passages between the two compartments are near the periphery, and have small apertures. The first disintegrator is of the swing-hammer or other yieldable type, and is surrounded by a grid, which is omitted at one part of the circumference, so that uncrushable particles may pass through into the space between the grid and the casing, and be collected in a pocket at the bottom. The second disintegrator is of a type more suitable for fine grinding, preferably comprising a number of fixed and rotating pins parallel to the axis. A fan produces an air-current which draws the fine material through the apparatus (against centrifugal force in the second disintegrator), and in the axial

outlet are additional centrifugal blades to catch coarse material and fling it back into the disintegrator compartment.

B. M. VENABLES.

Colloid mill. L. A. MOLIN (U.S.P. 1,650,088, 22.11.27. Appl., 17.12.25).—The mixture to be emulsified is supplied through a hollow shaft to the space between duplicate rotor elements on the shaft. Each element is provided with a set of emulsifying surfaces arranged at an angle to one another, and each set co-operates with a corresponding set of similar surfaces on an outer stator member to provide emulsifying clearances.

H. HOLMES.

Centrifugal machine. H. J. GREAVES (B.P. 280,363, 22.11.26).—The wall of a basket of a centrifugal machine is formed of a number of arc-shaped, pervious conveyors through which the liquid passes and on which the solid matter collects, and is conveyed to the end of the conveyors, where it flies out through suitable ports.

B. M. VENABLES.

Centrifugal machine. C. A. OLCOTT, ASSR. to S. S. HEPWORTH Co. (U.S.P. 1,652,908, 13.12.27. Appl., 3.6.25).—The lower edge of the basket wall is riveted to a peripheral flange of a bottom casting provided with ribs on its underside. A wind plate covers the ribs and overlaps the riveted edge and flange, and a reinforcing ring is shrunk on the overlapping portion.

H. HOLMES.

Centrifugal separator. G. TER MEER (U.S.P. 1,651,421, 6.12.27. Appl., 10.11.25. Ger., 12.3.25).—Spaced top and bottom walls and an axially movable peripheral wall are provided. The latter, adapted to seat against the top wall and to have a tight fit with the bottom wall, is fitted with a screen. Means are provided for moving the peripheral wall downwards to permit radial discharge of the material. A separating action is effected by a layer of previously separated material left on the screen.

H. HOLMES.

Circular-deck concentrator. A. H. STEBBINS (U.S.P. 1,650,725—6, 29.11.27. Appl., [A] 19.7.26, [B] 24.9.26).—A frame supports an annular deck provided with means for vibrating it. (A) The deck is frusto-conical, sloped to direct the tailings toward the central opening, and is provided with channels extending around it for more than a complete revolution upon a single deck surface. The forward and rear ends of the channels overlap upon a non-overlapping deck surface. (B) The deck is provided with a stepped spiral wall, of which one portion is close to the inner edge and the remainder diverges gradually therefrom. The materials are delivered to the deck near its inner edge. The concentrates are directed against the wall for re-treatment by riffles on the deck surface, and the tailings move outwards over the riffles.

H. HOLMES.

Spraying of liquids [or powders]. G. F. COURTOIS (U.S.P. 1,651,808, 6.12.27. Appl., 30.1.24. Belg., 5.2.23).—The material to be sprayed is contained in a receptacle into which extends a tube connected to the spraying nozzle. The receptacle is mounted concentrically within a telescoping casing, and a spring is enclosed in the space between them. This space communicates with the upper part of the receptacle, and a valve is provided for admitting air for compression within the space and the receptacle.

H. HOLMES.

Concentration of liquids. W. B. SENSEMAN (U.S.P. 1,651,557, 6.12.27. Appl., 17.4.26).—A thin film of the liquid is confined and supported at spaced intervals in a horizontal plane, and hot gases, projected upwards with a whirling movement against the unsupported portions, cause the film to rotate in its own plane.

H. HOLMES.

Apparatus for mixing powder with liquid. FOAMITE FIREFOAM, LTD., from FOAMITE-CHILDS CORP. (B.P. [A] 280,313, [B] 280,407, [C] 280,408, 1.9.26).—The liquid is passed through a supply conduit, Venturi tube, and delivery conduit, and the powder is drawn into the throat of the Venturi tube by the suction therein. (A) The Venturi tube converges first to a small throat, of which the length is rather greater than the diameter, then expands slightly to a throat of approximately double cross-section, of which the length is not less than twice the diameter, and finally expands gradually to the delivery conduit. The powder is supplied at or near the junction of the two throats into the larger one through a side passage, preferably inclined in the direction of liquid flow, which is not larger in cross-section than the throat itself. (B) A check valve is provided in the entry for powder adapted to be closed by the liquid, in case a positive pressure should develop instead of a vacuum. A light spring may be provided which tends to close the valve, so that it will only open if the suction is definite. The valve may be faced with rubber or any yielding metal, and the valve seat may consist of an annular, sharp edge. (C) In the delivery conduit a fixed guide of twisted form is placed to impart rotary motion to the stream of mixed powder and liquid.

B. M. VENABLES.

Apparatus for the dispersion of solids in liquids. W. H. WHATMOUGH (B.P. 280,973, 29.5.26).—The material is fed above the bite between two rollers which touch, but run upwards and carry the material under wiping blocks or bands, similar to those described in B.P. 276,400—1 (B., 1927, 831).

B. M. VENABLES.

Centrifugal extractor. E. J. CARROLL, ASSR. to AMER. LAUNDRY MACHINERY Co. (U.S.P. 1,652,760, 13.12.27. Appl., 25.1.23).—The spindle carrying the basket is mounted in spaced bearings on the main frame. Means are provided for preventing displacement of the bearings bodily while permitting their oscillation due to deflection of the spindle by an unbalanced load. The direct motor drive of the spindle includes a stationary part and a rotary part carried by the spindle, the clearance between these parts being sufficient to provide for the maximum deflection of the spindle at the motor.

H. HOLMES.

Centrifugal separation of substances. L. D. JONES, ASSR. to SHARPLES SPECIALTY Co. (U.S.P. 1,649,117—8, 15.11.27. Appl., [A] 7.10.21, [B] 24.12.23).—(A) The mixture is delivered to an inner zone at one end of the rotor and a heavier carrier liquid is delivered through an outer zone at the other end into the zone of separation. The bulk of the liquid is held in the rotor, and the separated constituents are discharged separately. (B) Part of the mixture delivered into the rotor is discharged, and an auxiliary is introduced at a point adjacent to the point of discharge, while maintaining the contents of the rotor in liquid balance.

H. HOLMES.

[Centrifugal] separation of liquids from solids.

H. C. BEHR (U.S.P. 1,650,685, 29.11.27. Appl., 12.12.25).—A foraminous shell and a controlling mechanism within it are mounted for rotation about their common axis. A slight difference of speed is maintained by a driving connexion between the shell and the mechanism including gearing engaging a fixed co-axial worm.

H. HOLMES.

Separation of solids from liquids in which said solids are suspended. A. E. WHITE. From A. WRIGHT and F. W. YOUNG (B.P. 280,389, 14.12.26).—A rotary-drum vacuum filter is provided, outside the actual filter medium, with a "reinforcement" for the cake to which the cake clings and by which it may be removed from the filter drum. The reinforcement may comprise mesh or similar material, but preferably comprises a number of parallel, endless wires wrapped round the filter drum and round another drum and system of rollers which may be used for cake removal and/or drying the material after filtration. The cake may be completely removed from the wires by means such as a comb or by causing them to pass round a roller in the form of a loop, the residue of cake being completely removed at the line of intersection.

B. M. VENABLES.

Hydraulic classifier and separating process.

F. W. SCHMIDT and W. M. GREEN (U.S.P. 1,650,239, 22.11.27. Appl., 14.9.23).—A liquid is added to the mixture and the whole is elevated and discharged. Oversize material is separated and washed with the discharged liquid, and the washings are combined with the main mass. Any proper-size material is removed from the oversize material by dry-screening.

H. HOLMES.

Filter press. M. KORFF, Assr. to J. P. BEMBERG A.-G. (U.S.P. 1,650,138, 22.11.27. Appl., 8.10.25. Ger., 9.10.24).—A filter disc with a releasable impervious sector is rotatably mounted in a casing, and an opening along one edge of the casing for removal and replacement of the disc is provided with a removable cover. Inlet and outlet apertures having liquid-tight engagement with opposite faces of the disc are provided in the casing remote from the cover, and are adapted to be closed by the sector while discs are being interchanged.

H. HOLMES.

Thickener. H. S. COE (U.S.P. 1,650,820, 29.11.27. Appl., 9.3.26).—A drag member extending across the settling area is attached to a tow line suspended from a driving element travelling around the area.

H. HOLMES.

Filter for fine filtration of liquids. O. PETERSEN (B.P. 280,802, 5.5.27).—A filter for lubricating oils etc. comprises a length of tube which is an exact fit inside a cup or bucket and has a number of holes drilled through both simultaneously. The two parts are then relatively rotated and pinned in position so that the holes in each element overlap only very slightly, giving the interstices which effect the filtration. The liquid is fed through a liquid-tight cap into the inner tube, the collected solids remain in the bucket, and the filtrate passes through into an outer casing.

B. M. VENABLES.

Filtering apparatus. W. E. BELKE (U.S.P. 1,652,423, 13.12.27. Appl., 11.9.26).—The filtering

medium is cup-shaped with the base of the cup detachably secured to a trough which receives the filtrate flowing down the outside of the cup and is provided with an outlet.

H. HOLMES.

[Material for] filters. J. HERRMANN and M. STAUBER (U.S.P. 1,649,405, 15.11.27. Appl., 24.4.24. Ger., 22.5.23).—Filtering material is prepared by shaping into coarse grains a silica gel containing less than 91% of water and drying the grains.

H. HOLMES.

Treatment of water, oil, or other liquids. J. CALDER (B.P. 280,991, 28.7.26 and 22.3.27).—When it is desired to introduce finely-divided solid matter, *e.g.*, metallic dust, into liquids, *e.g.*, boiler feed-water, the substance is reduced to the powdered form in the dry state, preferably by several stages of disintegration, and is then boxed to prevent oxidation. When required for use it is made into an emulsion with a portion of the liquid, the coarse particles are allowed to settle out or are screened out, and the fine emulsion only is added to the bulk of liquid in the container it is required to protect.

B. M. VENABLES.

Degassing of water. D. S. JACOBUS, Assr. to BABCOCK & WILCOX Co. (U.S.P. 1,650,129, 22.11.27. Appl., 13.6.19).—The greater part of the gases is expelled by boiling under partial vacuum, and the water is then passed over oxidisable material.

H. HOLMES.

Utilisation of liquids for effecting transfer of energy or matter or like purposes. SYNTHETIC AMMONIA & NITRATES, LTD., R. E. SLADE, A. F. BURSTALL, and W. F. CAREY (B.P. 279,526, 30.4. and 18.5.26).—The liquid is caused to flow in a thick, substantially uniform, turbulent film over a surface. To create and maintain the turbulence the liquid must flow at the rate of at least 300 c.c. per cm. of circumference measured transverse to the flow. The apparatus may be used for transferring matter (*e.g.*, scrubbing gases), for direct or indirect transfer of heat with or without evaporation or condensation, or the combined interchange of matter and heat known as rectification, and so long as the film is turbulent the transfer is rapid. The apparatus is preferably constructed of vertical tubes, and the films are formed by overflow of one or more liquids from an annular supply-channel and weir surrounding the inside, outside, or both sides of the tubes, or films may be formed down the inside of one and the outside of the other of a pair of tubes with a narrow annular space for the passage of gas or heating agent between the tubes. A multiple-effect evaporator may be constructed from a number of concentric tubes, heat from an external source being applied to the innermost only, vaporisation from turbulent liquid films taking place in alternate annular spaces outwardly, and successive condensation of vapours in the other alternate spaces.

B. M. VENABLES.

Method and apparatus for supplying fluids [in definite proportion]. A. E. WHITE. From GAS RESEARCH Co. (B.P. 280,010, 4.5.26).—An apparatus for supplying a number of fluids in predetermined definite proportions by volume, independent of density variations, comprises a number of centrifugal pumps delivering through orifices, one pair for each fluid. In the case of gas and air supplies to a continuous calorimeter, the air

will be drawn direct from atmosphere by one pump and delivered through an adjustable orifice comprising preferably a mushroom valve of which the stem is screwed and provided with a locknut. The gas, in general, will be supplied under slight pressure, which is neutralised by supplying too much gas to the pump suction chamber and allowing the excess to burn at an outlet freely open to atmosphere; the flame will afford an indication of whether the apparatus is working and of any change in the character of the gas. Both pumps are mounted on the same shaft. Since the amount of gas is smaller than the amount of air, and any deposition of tar or other matter on the orifice would have a serious effect on one small orifice, a number of larger orifices are used in series to get the same baffling effect. B. M. VENABLES.

Liquefaction of gases. G. F. JAUBERT (B.P. 244,468, 20.8.26).—The gas is compressed at a pressure at least equal to the liquefying pressure at the desired temperature, cooled by cooling water, and further cooled in a general heat interchanger. A portion of the gas is expanded in an engine and its temperature thereby lowered; this portion exchanges its cold with the unexpanded portion in a liquefier, the latter portion being thus liquefied. The expanded gas passes back through the heat exchanger to an intermediate stage of the compressor. When the liquefied gas is a mixed gas which it is desired to separate by rectification, the whole or part of the expanded gas from the engine, together with any vapour formed from the liquefied portion expanded to the same pressure, may be used to heat the lower part of the rectification column, thereby obtaining a certain amount of liquid in the heating coil. Part of the circulating gas may be used to re-compress and re-liquefy the volatile constituent from the top of the rectifier, in order to obtain pure washed liquids.

B. M. VENABLES.

Separating dust from gases. H. H. BUBAR (B.P. 261,706, 25.6.26. U.S., 20.11.25).—The dust-laden stream of gas is led horizontally through several ranks of vertical deflectors by which vortical motion is set up with downward axial progression. The progression delivers the dust into a space below where the vortices are broken up, the gas becomes quiet, and the dust is deposited.

B. M. VENABLES.

Removal of dust, sand, soot, and other foreign bodies from dust-laden air, flue gases, etc. J. THOMPSON (DUDLEY), LTD., and R. D. WEST (B.P. 281,033, 10.9.26).—The gas is impelled by a fan, and in the fan casing is a skimming plate which takes off the outer layer of air most highly charged with dust, which is then separated in a small separator. The larger stream or the recombined streams may pass through a large separator of simple form in the final outlet.

B. M. VENABLES.

Treatment of gases, fumes, etc. G. S. PURTYMAN (U.S.P. 1,652,956, 13.12.27. Appl., 28.9.25).—A main chamber is attached at one end to the base of a conical chamber, and each chamber is provided with spraying devices and with a floor formed in inclined sections. Gas inlet means conforming to the shape of the floor are provided in the main chamber and gas outlet means in the conical chamber.

H. HOLMES.

Dehydration and kindred treatment [selective adsorption] of gases, combustion products, vapours, etc. J. A. REAVELL (B.P. 280,268, 6.8.26).—In an apparatus for the removal of a vapour or other constituent of a gas by selective adsorption in solid absorbents such as silica gel, the absorbent in finely-powdered form is sprayed, preferably centrifugally and horizontally, in the upper part of a container in which the gas passes downwards, preferably with a whirling motion. The solid absorbent is deflected by baffles into the bottom of the vessel, whence it is removed for regeneration and re-use, and the gas passes through a side outlet to a cyclone or other separator for the removal of last traces of solid absorbent.

B. M. VENABLES.

Air classifier. A. H. STEBBINS (U.S.P. 1,650,727, 29.11.27. Appl., 28.9.26).—A mixture of fine dust-like particles to be classified is delivered into the upper end of a tall casing divided into superposed compartments, and a group of deflectors is mounted in each compartment for dispersing and retarding the falling particles. The heavier particles pass through the successive chambers, and the lighter particles are withdrawn by currents of air.

H. HOLMES.

Apparatus for effecting chemical reactions. H. HENNEBUTTE and E. GOUTAL (U.S.P. 1,650,713, 29.11.27. Appl., 21.5.23. Fr., 7.6.22).—The material under treatment is propelled by endless screws through a series of troughs and is forced by distributors from one trough to the next. The troughs are provided with convex covers having peripheral gutters extending inwards, and discharge openings communicate with the lowest points of the gutters.

H. HOLMES.

Radiation pyrometers. A. SCHWARTZ (B.P. 280,454, 27.5.27).—In a pyrometer of the type where the concentrated radiation falls on a bi-metallic spiral which moves a pointer, the scale also is attached to a bi-metallic spiral, screened from the radiation but affected by the room temperature and moving the zero of the scale in accordance with the latter. Arrangement is made to illuminate the scale by the luminous rays from the source of radiation being measured.

B. M. VENABLES.

[Short-flame, pulverised fuel burner for boiler] furnaces. GEN. ELECTRIC CO., and G. G. BELL (B.P. 280,634, 17.8.26).

Regeneration of catalysts (B.P. 280,712).—See VII. **Tunnel kilns (B.P. 280,044).**—See VIII. **Evacuation of vessels (U.S.P. 1,651,386).**—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Conversion of lignite and coal into liquids and oils. W. A. DYES (Chem.-Ztg., 1927, 51, 853—855, 873—876).—Synthetic methyl alcohol is too expensive to replace gasoline at present prices. Motor spirit obtained from coal and its products by the Bergius process has also to compete with the relatively cheap petroleum product. The cost of berginisation depends largely on that of hydrogen and on the use of suitable catalysts. Light oils are obtainable from tar products by cracking, alone or accompanied by hydrogenation. The various methods of obtaining oils from lignite and coal are surveyed from a commercial point of view, with

particular reference to conditions in Germany and to the work of the I.-G., and detailed references are made to the relevant patent literature.

W. T. K. BRAUNHOLTZ.

Lignin and oxycellulose theories [of coal formation]. J. MARCUSSEN (*Z. angew. Chem.*, 1927, **40**, 1233—1234).—Analyses of three lignites are given, in two of which the lignin content (28—30%) is the same as that of the original conifer wood, so that their contents of free humic acids, humic acid anhydrides, and ketones (22% and 41% respectively) must be ascribed to cellulose originally present. The third lignite contains 0.9% of lignin, 33% of free humic acids, and 15.3% of cellulose present almost entirely as oxycellulose. Further evidence that humic acids and coal are, in part, derived from carbohydrates is given by the oxidation of humic acid with hydrogen peroxide, whereby a good yield of a solid, brownish-black acid, called "caramelic" acid (from its odour), is obtained, the same acid being also obtained by treating grape sugar in the same way.

W. T. K. BRAUNHOLTZ.

Horizontal settings, with special reference to silica retorts. S. HAY (*Gas J.*, 1927, **180**, 538—543, 612—616, 808—812).—Continuous observations for five years show the advantages of using silica segmental retorts when the average combustion chamber temperature is as high as 1325° and the retort temperature about 1050°; these conditions are above the working limit of fireclay or siliceous material, and retorts made of 95% silica have better thermal conductivity, increased throughput, and greater flexibility. The general use of silica in producer construction, except for the nostril blocks, is not recommended, but owing to the allotropic changes undergone by silica at 230°, all-silica construction of the setting is essential to ensure uniform expansion. Suitable silica ware should have d 2.35 (approx.) and its porosity should be below 27%, or else increased slag and scurf penetration and spalling will occur. Failures are due not to high temperatures alone, but to such other causes as faulty design, mechanical crushing, inferior jointing or patching cements, and iron attack, so that particular care in the selection of materials is necessary. A setting containing both moulded siliceous retorts and those of 95% silica has been examined in detail after use, and the superiority of the latter is clearly demonstrated.

[With A. EDWARDS.] Failure of silica retorts is frequently due to the use of unsuitable cements for jointing or repair, and existing faults may extend rapidly if badly repaired. Examination of a large number of cements shows that a high silica content is not the sole criterion of satisfactory behaviour, but that a low percentage of alumina is always desirable. The life of a jointing material will depend largely on its reaction with iron compounds; these are undoubtedly derived from the coal, and are remarkably destructive on account of slag formation. A large number of specimens discoloured or slagged to different extents have been analysed, and the selective absorption of ferric and ferrous oxides, and not of the ash constituents as a whole, by cementing materials has been demonstrated; it is this absorption proceeding from inside the retort which lowers the m.p. so that failure ultimately occurs. The behaviour of a

number of special cements has been tested at high temperatures under load; improvements due to substitution of lime instead of alumina are noticeable. A jointing material made up with glue behaves extremely well in use although its application is troublesome. It is suggested that failures would be reduced in number by adoption of combustion chamber design more closely resembling that used in the case of coke ovens, where less extreme local temperatures are necessary.

R. H. GRIFFITH.

Carbonisation in vertical retorts. T. C. FINLAYSON (*Gas J.*, 1927, **180**, 679—686).—The principles which have governed the development of the Woodall-Duckham continuous vertical retorts are discussed, particular consideration being given to (1) the coke extraction device, (2) top ironwork design, (3) taper of retort, (4) method of heating, and (5) retort construction. Among the advantages of carbonisation in such systems are the large output of gas per sq. ft. of ground space, high yield of gas per ton and flexibility of the system due to the facilities for steaming, high thermal efficiency, and low capital cost. The coke formed is particularly suitable for domestic use. A method of scurfing retorts by means of water-gas production, the W.-D. swelling number test, and the use of intermittent vertical chambers are briefly discussed.

A. B. MANNING.

Vertical intermittent chamber ovens for gas manufacture. N. J. BOWATER (*Gas J.*, 1927, **180**, 686—688).—The recent extension of the use of intermittent vertical retorts in Germany is due principally to the wider range of choice of coal thereby permitted. Other advantages over the continuous system lie in the simpler charging and discharging apparatus necessary, and the smaller proportion of breeze formed. The mode of operation of a setting of intermittent vertical retorts is briefly described, and the results of a test carried out on a Durham coal (Londonderry) are summarised.

A. B. MANNING.

Partial drying of town gas. C. HARRIS (*Gas J.*, 1927, **180**, 529—535).—Taunton town gas is partially dried by Messrs. Holmes & Co.'s "Dri-Gas" process. The installation comprises a rotary brush scrubber washer in two sections, each of six chambers, the first six being supplied with a 40% calcium chloride solution for gas drying, and the second six with gas oil for removal of naphthalene. The drying solution is kept circulating through a cooler, about 5% being by-passed over an evaporator to maintain the total solution at constant strength. The reduction in the dew-point of the gas is about 30° F., which is raised again about 2—4° F. in the gas holder, the water in which carries a film of oil. Practically complete elimination of naphthalene is attained by the use of about 14 gals. of gas oil per million cub. ft. of gas. The total cost of the process is 0.23d. per 1000 cub. ft. The advantages accruing from the partial drying of gas include the prevention of corrosion in mains and meters, the possible use of steel mains and welded joints, the reduction in maintenance costs, more efficient elimination of naphthalene, and a greatly improved service to consumers by eliminating stoppages. The moisture content of the gas is determined by passing it over a weighed amount of calcium chloride, or, more

rapidly, by determining the dew-point by circulating the gas round a tube containing ether which is gradually cooled by evaporation.

W. T. K. BRAUNHOLTZ.

Origin and decomposition of carbon disulphide in gas-making. The carbon-sulphur complex. W. J. HUFF and J. C. HOLTZ (Ind. Eng. Chem., 1927, 19, 1268—1271).—By cracking gas oil of high sulphur content (3.61% S by wt.) at temperatures between 649° and 982°, it was observed that raising the temperature increased the yield of carbon disulphide, but no information was obtained as to whether the carbon disulphide was formed by the decomposition of complex sulphur compounds or by synthesis from carbon and sulphur. Several litres of sulphur-free oil gas were therefore prepared by cracking a medicinal oil. These were mixed with hydrogen sulphide (0.2 g./m.³), and the mixture was passed through the clean cracking tube while additional medicinal oil was cracked therein. No carbon disulphide was formed, but a loss of hydrogen sulphide took place. Carbon disulphide appeared when a much higher concentration of hydrogen sulphide (69 g./m.³) was used. A carbonaceous deposit always appeared in the cracking tube, and the formation of carbon disulphide may have been due to reaction of the hydrogen sulphide either with this or with the cracked oil products. Nitrogen mixed with hydrogen sulphide (4.6 g./m.³) was passed over sulphur-free sugar charcoal at the cracking temperature (843°), and an extended delay occurred before hydrogen sulphide appeared in the issuing gases. No carbon disulphide was detected until about 20 litres of gas had passed over the charcoal. The latter finally contained 2.8% S. It is suggested that the formation of a solid sulphur-carbon complex is an intermediate stage in the production of carbon disulphide.

R. C. ODAMS.

Processes involving the carbonisation of coal by internal heating. M. W. TRAVERS (Gas J., 1928, 181, 36—38).—From the thermal analysis of the changes taking place in the fuel bed of the gas producer operated under varying conditions, information can be obtained in connexion with the gasification of solid fuel by "internal heating." Equations for compiling a thermal balance-sheet are given and applied to determine whether a process involving the gasification and carbonisation of coal in two stages is possible. The method of investigation is applied to the low-temperature carbonisation of coal by producer gas or by steam, and the complete gasification of coal to produce mixed water-gas and coal gas.

H. S. GARLICK.

Examination of phenols present in tar from the wood of Swedish conifers. P. KLASON and H. MELLQUIST (Z. angew. Chem., 1927, 40, 1231—1233).—The tar was fractionally distilled at 30 mm. pressure, and the fractions were extracted with 10% potassium hydroxide. The phenols and acids obtained by acidifying the extract were methylated with methyl sulphate, and the acids removed by hydrolysing the methyl esters produced. The methylated phenols remaining were fractionally distilled and examined. The tar contains *m*- and *p*-cresol (no *o*-cresol), guaiacol, creosol, ethyl-guaiacol, and either propylguaiacol or *isoeugenol*, creosol preponderating.

W. T. K. BRAUNHOLTZ.

Deterioration of mineral oils. II. Mechanism of oxidation and action of negative catalysts as determined by static methods. (a) B. MEAD, A. C. PHELPS, C. R. WASHBURN, and J. P. WARNER; (b) B. MEAD, E. F. DIRKS, and W. B. BADER; (c) B. MEAD and W. L. MCCABE (Ind. Eng. Chem., 1927, 19, 1240—1245; cf. B., 1927, 355).—Static methods have been found preferable to dynamic methods where the experiment was of long duration or when a large number of samples were to be studied. (a) *Oxidation by closed static method.*—The oil and oxygen were contained in a closed apparatus, so designed that the amount of oxygen absorbed by the oil could be determined by actual measurement. The oil was heated at 130° in an oil bath designed to maintain the temperature constant within 0.5°. This temperature was chosen, as experiments at higher temperatures gave evidence of a change in the mechanism of sludge formation above 140°. The rate of oxygen disappearance at 120° and at 130° was found to be directly proportional to the time of exposure of the oil. The rate of absorption at the higher temperature was 2.4 times as great as that at the lower temperature; this figure would be much lower if diffusion of oxygen into the oil were the controlling factor of the oxidation. The amount of surface of oil exposed to oxygen (0.2—0.4 cm.²/g.) had no measurable effect on the rate of oxygen disappearance at 120°, but at higher temperatures may exert some influence. Sludge formation and acidity at first developed very slowly, but their rate of formation increased as the experiment progressed. The development of acidity may have a catalysing action upon sludge formation. The rate of oxygen disappearance seemed also to depend upon the degree of chemical unsaturation of the oil; this relation held specially well for oils from the same base. (b) *Oxidation by open static method.*—The samples of oil were heated in open test tubes at 150° in an oil bath contained in an air oven. Acidity development was autocatalytic only when the oil had a low initial acid value. High initial acid values were obtained by the addition of deteriorated oil (up to 5%). Sludge formation was much greater when the initial acid value of the oil was high. (c) *Prevention of deterioration by use of antioxidants.*—A definite volume of the oil containing the antioxidant (0.15%) was placed in an open test tube, suspended in an oil bath, and heated at 130—140° for 130—168 hrs. The sludge was removed by filtration through a Gooch crucible, washed with light petroleum, dried to constant weight at 110° in an air oven, and weighed. Of the 177 substances tested, 48 showed an inhibiting effect on sludge formation. Sulphur, nitroresol, and nitrobenzene had by far the greatest effect. No generalised conclusions could be drawn as to the types of compounds which acted as anticatalysts.

R. C. ODAMS.

Determination of sulphur and sulphur derivatives of hydrocarbons in naphtha solutions and in petroleum distillates. W. F. FARAGHER, J. C. MORRELL, and G. S. MONROE (Ind. Eng. Chem., 1927, 19, 1281—1284).—In a naphtha solution containing hydrogen sulphide, sulphur, mercaptans, organic sulphides, disulphides, and thiophen, the hydrogen sulphide was determined by shaking for 2—3 min. with an acidified solution of cadmium chloride (10 g. in 100 c.c. of water,

with 1 c.c. of hydrochloric acid). The percentage of hydrogen sulphide present was calculated from the weight of cadmium sulphide formed. No cadmium mercaptides were precipitated. The layer of oil was removed, the sulphur content determined by the lamp method, and the oil shaken with metallic mercury. The mercury sulphide was filtered off, and the sulphur content of the oil again determined, the difference being the amount of elementary sulphur present. The remaining oil was divided into two parts (a) and (b). Mercaptans were determined in (a) by dissolving the oil in benzene, and shaking either with alcoholic sodium plumbite solution, or with an aqueous suspension of basic lead acetate. In the former case the lead mercaptides formed dissolved in the alcohol, and a lamp determination on the oil gave by difference the sulphur removed. In the latter the aqueous layer was removed and the benzene layer, containing lead mercaptides, agitated with a known quantity of 0.1*N*-sulphuric acid. Lead sulphate was collected on a filter and the acid remaining in the solution and washings titrated with 0.1*N*-sodium hydroxide. The sulphur present as mercaptans was calculated from the acid consumed. The portion (b) was reduced with zinc and dilute acid (10%) and the disulphides present were reduced to mercaptans, which were extracted with alcoholic plumbite, and the remaining sulphur was determined by the lamp method. The amount of sulphur present as disulphides was given by the difference between the mercaptan sulphur previously determined and the combined disulphide and mercaptan sulphur. The oil was next treated with mercurous nitrate to remove sulphides, and the residual sulphur determined by the lamp method, the sulphur present as sulphides being determined by difference. The residual sulphur represented thiophen which was not removed by any of the reagents used. The method was applied to various petroleum distillates. Californian cracked gasoline contained 1.1% S (86% residual); Smackover cracked gasoline 0.2% S (80% residual); and straight-run Panhandle gasoline 0.09% S (elementary and mercaptan sulphur).

R. C. ODAMS.

Determination of aromatic and unsaturated hydrocarbons in petroleum spirit by means of sulphuric acid. R. KATTWINKEL (Brennstoff-Chem., 1927, 8, 353—358).—Addition of phosphorus pentoxide to concentrated sulphuric acid increases its reactivity so that it readily sulphonates the aromatic compounds in the cold, whilst addition of boric acid reduces the reactivity of the acid sufficiently to enable it to be used for determining the content of unsaturated hydrocarbons. The content of aromatic and unsaturated hydrocarbons together is determined by shaking 10 c.c. of the benzine with 30 c.c. of the acid mixture (prepared by dissolving 30 g. of phosphorus pentoxide in 100 c.c. of sulphuric acid, *d* 1.84) for 5 min., the benzine being diluted with a standard benzine in cases where the temperature rises above about 40°. After leaving the mixture to settle, the increase in volume of the acid is noted. The unsaturated hydrocarbons alone are similarly determined by shaking the benzine with an acid mixture prepared by dissolving 5 g. of boric acid in 100 c.c. of sulphuric acid (*d* 1.84).

W. T. K. BRAUNHOLTZ.

State of asphaltenes and resins in petroleum and its products. A. SACHANEN (SACHANOV) and N. WASSILIEV (Petroleum, 1927, 23, 1618—1621; cf., B., 1925, 745).—The method of preparation of petroleum resins previously described has been altered in order to prevent their conversion into asphaltenes; naphthenic and asphaltogenic acids are removed from the petroleum with caustic soda, and asphaltenes are precipitated by light petroleum. The residue is treated with silica gel at 50° in amounts determined by the resin content, and unchanged oil is extracted with benzene. Neutral resins from a petroleum, various distillates from it, a tar, and an asphalt were compared in yields, physical properties, etc., and elementary analyses are given. Mol. wt. determinations carried out in benzene show an increase at higher concentrations, values between 290 and 788 being found; the mol. wt. of asphaltenes are about 5000—6000. Neutral resins thus form a series of oxidation products, but not condensation products, from hydrocarbons, and have the formula $C_nH_{2n-m}O_2$, where *m* varies from 9 to 33, suggesting a highly polycyclic structure which is only slightly unsaturated. Although unstable, the resins are easily distilled at low pressure; they show normal temperature-solubility curves with no evidence for colloid formation. They are readily converted into asphaltenes by oxidation, which takes place by removal of hydrogen, accompanied by further condensation. Sulphuric acid acts, sometimes slowly, to give asphaltenes and sulphonation products.

R. H. GRIFFITH.

Action of refining agents on pure [organic] sulphur compounds in naphtha solution. M. A. YOUTZ and P. P. PERKINS (Ind. Eng. Chem., 1927, 19, 1247—1250).—The action of various desulphurising agents upon pure organic sulphur compounds dissolved in naphtha has been studied, solutions of such strength being used that each contained 0.4—0.6% S. Sulphuric acid (95%) had chiefly a solvent action. Aluminium chloride in limited quantity removed all compounds except ethyl sulphide and *n*-heptyl sulphide. Ethyl disulphide was decomposed with formation of hydrogen sulphide and mercaptan. Silica gel lowered the sulphur content of all the solutions, the least decrease being observed with thiophens, disulphides, and tetramethylethylene sulphide. Fuller's earth at 400° removed all sulphur compounds fairly well, with the exception of thiophens. Mercuric iodide in conjunction with methyl iodide extracted aliphatic sulphides very readily from solution in petroleum fractions of high or low mol. wt.

R. C. ODAMS.

Treatment of benzine obtained by cracking processes and by low-temperature carbonisation [of lignite] with sulphuric acid and liquid sulphur dioxide. A. ERDÉLY and L. ALMÁSI (Brennstoff-Chem., 1927, 8, 358—360).—Benzine produced by cracking Rumanian gas oil and lignite producer tar oil and fractionally distilling the products up to 200°, and by fractionally distilling lignite low-temperature tar up to 200°, was examined by the method of Riesenfeld and Bandte (Erdöl u. Teer, 1927, 3, 139). The corresponding fractions from the two cracked products were similar in composition (*d* 0.680—0.828, with, roughly, 12—35%

of unsaturated compounds, 4–20% of aromatic compounds, 24–38% of naphthenes, and 20–38% of paraffins), but differed from those from the low-temperature tar (d 0.816–0.851, with, roughly, 46–55% of unsaturated compounds, 14–22% of aromatic compounds, 15–18% of naphthenes, and 14–15% of paraffin). The oils do not react with sulphuric acid of less than 40% strength, and acid of 85% strength dissolves twice as much as 70% acid. The volume of 85% acid required to remove all the acid-soluble components is $1\frac{1}{2}$ –2 times that of the oil treated. Shaking the oils with twice their volume of 85% acid reduces the iodine value of the undissolved residue to about half that of the original product, whilst a similar volume of 100% acid reduces it from 73–160 to 7–15. Sulphuric acid not only dissolves certain components of the oils, but causes polymerisation, forming products which are not extracted from the benzene even by 100% acid. The action of liquid sulphur dioxide on the cracked products did not effect a complete separation of saturated from unsaturated and aromatic hydrocarbons, whilst with the lignite tar-oil benzene the mixture did not separate into two layers at -12° . The smaller the proportion of unsaturated and aromatic compounds present, the more complete is their separation from saturated hydrocarbons by liquid sulphur dioxide.

W. T. K. BRAUNHOLTZ.

Control of lubricating oil refining. E. GRUNEWALD (Petroleum, 1927, 23, 1621–1622).—Chemical and physical methods of refining lubricating oils produce results which are determined by measurement of viscosity, colour, etc. In order to get some numerical idea of the effect, a colour scale of 26 degrees has been constructed by mixing water-white oil with heavy oil in different proportions and mounting these samples in vessels of standard size. The difference in degree of colour is considered simultaneously with alteration of other properties.

R. H. GRIFFITH.

Evaluation of watch and clock oils. P. CUYPERS (Chem.-Ztg., 1927, 51, 841–842).—A content of 2–3% of free fatty acid has a corrosive effect on the highly polished steel pivots and bearings of watches, and any copper, zinc, or nickel hastens the hydrolysis by catalytic action. Fatty oils alone are unsuitable, since they are not so stable as mineral oils owing to oxidation, polymerisation, and rancidity, and their viscosity and free fatty acid content increase in contact with air. In general practice, a mixture of mineral oil (liquid paraffin) with 50–60% of neatsfoot oil is used; the latter consists mainly of esters of fatty acids and withstands a temperature of -20° , whilst the mineral oil improves the chemical stability of the neatsfoot oil. In the evaluation of such lubricants the acid value and degree of rancidity are of special importance, and free alkali should be tested for, since the gradual saponification of an oil is more harmful than the presence of free fatty acid. W. G. CAREY.

Manufacture of hydrogen. LOSCHKAREV. **Caking of ammonium sulphate.** ADAM.—See VII. **Swelling of bentonite.** DAVIS.—See VIII.

PATENTS.

Low-temperature carbonisation process. METALLBANK & METALLURGISCHE GES., A.-G., Assees. of O.

HUEBMANN (G.P. 443,319, 18.1.24. Addn. to G.P. 440,379; B., 1927, 960).—The procedure of the main patent is modified by removal of the tar from the gas added to the main circulating current, before the latter is used for cooling the coke and for heat recovery. Condensation of tar on the coke is thus avoided.

A. B. MANNING.

Manufacture of fuel briquettes. A. M. HART (B.P. 279,140; 22.4.26).—Sodium or potassium silicate is mixed with finely-divided vegetable matter, *e.g.*, grasses, leaves, etc., and moulded into briquettes, which are then dried or heated until hardened. The vegetable material can also be first carbonised and mixed with other carbonised products, such as from coal or lignite, prior to mixing with the silicate and briquetting. The silicate (3–7.5%) is added in the form of powder or as a solution.

A. C. MONKHOUSE.

Preparation of water-gas and other combustible gases. I. G. FARBENIND. A.-G., Assees. of F. WINKLER (G.P. 443,445, 7.5.25. Addn. to G.P. 437,970; B., 1927, 548).—Air, preferably at a high temperature, is blown into the hot dust-laden gases leaving the fuel layer in the process described in the main patent in such amount as to gasify the solid fuel present and leave only an inconsiderable residue. A. B. MANNING.

Manufacture of mixed water-gas and coal gas. W. D. WILCOX (U.S.P. 1,652,386, 13.12.27. Appl., 20.3.22).—A fuel bed in one of two gas generators is raised to incandescence by means of an air-blast, and the blast gases are burnt in (and thereby used to heat) a superheater having a baffle wall. The air-blast is now shut off and steam is admitted to the base of the incandescent fuel, the resulting water-gas (after heating by passage through the superheater) being passed downwardly through another gas generator containing a fuel bed having an upper layer of coal, which thereby undergoes distillation with the production of a mixture of water-gas and coal gas. The operation of the gas generators is periodically reversed. C. O. HARVEY.

Production of fuel gases. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 279,316, 22.3.27. Addn. to B.P. 214,544; B., 1924, 549).—Finely-divided fuel is fed continuously into the side of a producer by means of a worm-conveyor, and the whole bed of fuel is kept in violent agitation by preheated air blown in for the combustion of the fuel. Any fuel carried forward as dust is separated from the gas and may be returned to the producer. When water-gas is being made and fuel is continuously being fed to the producer, a water-gas of high calorific value is obtained.

A. C. MONKHOUSE.

Apparatus for purifying coal gas. G. CHRISP (B.P. 279,184, 28.7.26).—Gas is purified by passage through two series of horizontal tubes enclosed in steam or hot water jackets and containing iron oxide which is fed along the tube in counter-current flow by a worm-conveyor. A third series of horizontal tubes, cooled if necessary, contain spent oxide which is being revived by air prior to use again. The oxide is fed from a hopper through a gas-tight valve, and is raised by elevators from one series of tubes to another. The worms of the conveyor are geared together, and the oxide can be

controlled by the speed of the elevators, the rotary valves, and the worms. A. C. MONKHOUSE.

Wash-oil for the recovery of light oil from gases.

I. G. FARBERIND. A.-G., Assecs. of W. SCHÄFER (G.P. 444,529, 26.6.25).—The mineral oil constituents soluble in liquid sulphur dioxide, which form an almost valueless by-product of the Edeleanu process, can be used as a gas-washing oil for the recovery of light spirits.

A. B. MANNING.

Gas calorimeter. J. F. SIMMANCE (B.P. 279,253, 3.11.26).—The recording mechanism of the differential thermometer of the gas calorimeter (cf. B.P. 115,915 and 189,665; B., 1918, 447 A; 1923, 123 A) is mounted so that it can be readily removed and adjusted without interfering with the remainder of the apparatus. Cold water after passing through a filter flows into a vessel with a weir overflow and from there through an orifice to the differential thermometer. The tube with the orifice can be removed for cleaning, during which period a cap with a similar orifice is fitted to the vessel.

A. C. MONKHOUSE.

Distillation systems [for benzolised oil]. SEMET-SOLVAY Co., Assecs. of A. F. KEANE (B.P. 269,517, 1.4.27. U.S., 17.4.26).—Light oils are recovered from a benzolised wash-oil by distilling in a vertical still comprising a series of ring sections, each containing a hood to give intimate contact between vapour and liquid, and alternate rings are staggered to give a circuitous path to the liquid. Steam is admitted at the bottom of the still and the vapours pass from the top of the still to a single-unit heat exchanger and condenser. The vapours enter the bottom of the heat exchanger and pass through a series of chambers, each containing coils through which flow the incoming benzolised oil, which is thus preheated to 95°. The vapours then pass to the top of the condenser situated beneath the heat exchanger, and are cooled in chambers containing coils through which cold water flows. The condensed oils pass to a separator where the water is separated. A. C. MONKHOUSE.

Cracking of [mineral] oils. W. BRINK (U.S.P. 1,652,344, 13.12.27. Appl., 3.3.25).—The oil is cracked at temperatures varying from about 246° to 526° (according to the grade of oil under treatment) and at pressures of about 50 lb./sq.in. The process involves continuous passage of the oil through a heating zone, and thence to a chamber containing a baffle, whence the vapours pass to the condenser and the unvaporised residue is allowed to drip into a large cylindrical chamber having a hemispherical base. The rate of flow of the oil is thereby checked and the precipitated carbon collects at the base of this chamber, the oil for retreatment overflowing into the heating zone. C. O. HARVEY.

Cracking of hydrocarbons. E. W. ISOM, ASSR. to SINCLAIR REFINING Co. (U.S.P. 1,650,519, 22.11.27. Appl., 28.1.22).—The vapours obtained by cracking the oil under pressure are passed up a reflux tower along with a stream of cool hydrocarbon gases. Fresh cracking stock passes down the tower in direct contact with the vapours and the admixed fresh stock and reflux condensate are passed into the still. C. O. HARVEY.

Hydrogenation and cracking of hydrocarbon compounds. INTERNAT. BERGIN-COMP. VOOR OLIE- EN

KOLENCHÉMIE, and A. DEBO (B.P. 280,734, 10.1.27).—In a process for increasing the yield of benzine vapours obtained during the cracking and hydrogenation under pressure of coal, oil, and other hydrocarbon compounds and for utilising the compression energy of the residual hydrogenating gas, the material to be treated is heated with hydrogen in a chamber maintained at a pressure of about 150 atm. and the products of the destructive distillation are cooled to about 300° and allowed to expand to a pressure of about 100 atm. Separation of "middle oil" takes place in a second cooler, whence the mixture of water and benzine vapours and permanent gases passes by way of a third cooler to a scrubbing tower, down which flows the "middle oil" previously condensed. The scrubbed gases are utilised, preferably after passage through a heater, to actuate an expansion engine, and pass thence to a methane-cracking device. C. O. HARVEY.

Manufacture of liquid or other hydrocarbons or derivatives thereof from carbonaceous materials. I. G. FARBERIND. A.-G. (B.P. 257,912, 18.8.26. Ger., 2.9.25).—Coal, tars, mineral oils, etc. which contain objectionable sulphur compounds are given, prior to destructive hydrogenation, a preliminary treatment under pressure and at temperatures above that at which decomposition commences with water or water vapour with or without the addition of hydrogen and preferably in the presence of catalysts, whereby the sulphur present in the original material is converted into hydrogen sulphide, which dissolves in and is removed with the water.

C. O. HARVEY.

Conversion of petroleum oils. C. P. DUBBS, ASSR. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,652,166, 13.12.27. Appl., 16.2.22).—The oil is simultaneously dehydrated and cracked by passage down a dephlegmating column up which pass the hot vapours from an enlarged reaction chamber which is connected with a cracking still. After withdrawal of water vapour, the oil passes to the cracking still. The vapours undensified in the dephlegmator are collected.

C. O. HARVEY.

Treatment of hydrocarbon oils. C. P. DUBBS, ASSR. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,652,167, 13.12.27. Appl., 2.5.23. Renewed 10.3.27).—The oil is cracked under a pressure of about 50 lb./sq. in. by passage through a relatively small coil to a larger vapour-generating coil, both coils being enclosed in a heating zone. Vapours are drawn off from the larger coil, whence the oil circulates back to the smaller coil, thence again to the larger coil, and so on continuously.

C. O. HARVEY.

Process and apparatus for the refining or cracking hydrocarbons. R. K. COLLINS (B.P. 280,034 and 280,039, [A] 12.11.26, [B] 16.11.26).—(A) The vapours from a horizontal still pass through a dome and pipe line fitted with a non-return valve to a specially constructed expansion chamber heated by a furnace or by the products of combustion from the still furnace. The vertical, cylindrical, expansion chamber is provided with a number of pipes (slightly staggered from the vertical) through which the hot flue gases pass, and around which the hydrocarbon vapours circulate in an

upward direction. These pipes may be wired and used as electrical heating elements. Nickel baffle plates are provided to check the upward course of the vapours undergoing treatment, and it is claimed that the method of heating minimises the formation of carbonaceous products by causing the vapours in immediate contact with the heating surfaces to circulate outwardly away from these surfaces. (b) Structural improvements relating to the above apparatus are described.

C. O. HARVEY.

Fractionation of hydrocarbon oils. J. C. BLACK (U.S.P. 1,652,886, 13.12.27. Appl., 5.4.23).—Vaporised hydrocarbons are partially condensed in a tower and the condensate is cooled and used partially to condense the residual vapours. Finally the ultimate residual vapours are condensed.

C. O. HARVEY.

Distillation of oil. G. W. WALLACE and H. K. IHRIG (U.S.P. 1,650,169, 22.11.27. Appl., 17.9.23).—The oil is cracked in a still provided with a number of pairs of heated tubes, each pair being traversed by an endless device carrying a catalyst, whereby the oil is continuously circulated through the tubes in contact with the catalyst, and carbonaceous deposits are prevented from adhering to the tubes.

C. O. HARVEY.

Treatment of hydrocarbons. C. S. CORNING, Assr. to W. S. CORNING (U.S.P. 1,652,394, 13.12.27. Appl., 8.9.23).—The hydrocarbons of lower b.p. are removed from a hydrocarbon mixture without substantial cracking by passing in thin sheets through retorts contained in a furnace. The vapours from each retort are subjected independently to a cracking treatment and are then blended.

C. O. HARVEY.

Reclaiming waste mineral lubricating oil containing soaps. H. A. FRASCH (U.S.P. 1,651,688, 6.12.27. Appl., 27.6.21).—Waste oil containing oleates is treated with sulphuric acid, and the resulting mixture of oil and oleic acid is separated from insoluble matter. After treatment at 50–60° with a quantity of an alkaline saponifying agent sufficient for neutralisation of the oleic acid, the oil-soap mixture is again separated from oil-insoluble matter.

C. O. HARVEY.

Manufacture of lubricants. O. ROLFSEN (B.P. 258,606, 17.9.26. Norw., 18.9.25).—A lubricant suitable for internal-combustion engines etc. consists of a mixture of a mineral oil with 0.5–10% of a marine-animal oil (*e.g.*, whale oil) previously hydrogenated under pressure and in the presence of a catalyst.

C. O. HARVEY.

Refining of mineral oils etc. P. BORNKESSEL (G.P. 442,893, 3.1.26).—The oil enters tangentially the lower part of the vessel containing the refining agent (acid, caustic soda, or water) so that the contents are given a circular motion. The oil rises spirally through the refining agent and leaves the vessel at an outlet in the upper part. The process avoids the use of stirring apparatus in the refining vessels.

A. B. MANNING.

[Refining of mineral oil by] treatment of one fluid by another. S. J. DICKEY, Assr. to GEN. PETROLEUM CORP. OF CALIFORNIA (U.S.P. 1,652,399, 13.12.27. Appl., 16.1.23).—An apparatus for the continuous treatment of oil with a relatively heavier liquid reagent consists of a cylindrical chamber to which the oil is

supplied under pressure, and in which admixture with, and separation from, the reagent occurs, the treated oil passing out continuously from the top of the chamber.

C. O. HARVEY.

Catalytic production of hydrocarbons from oxides of carbon and hydrogen. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 279,347, 30.5.27).—In processes of the above types the reaction tubes are arranged inside a high-pressure boiler. The temperature of the reaction is controlled and a heat exchange is effected.

A. C. MONKHOUSE.

Fuel for internal-combustion engines. CONTINENTALE A.-G. F. CHEMIE (B.P. 267,079, 12.8.26. Ger., 8.3.26).—“Knocking” in the internal-combustion engine is stated to be caused by the high insulating power of pure benzines which admits of the development of local electrical tensions in the compressed vapours with consequent pre-ignition. To increase the conductivity of the fuel, salts of heavy metals (*e.g.*, copper nitrate) are added, a solution being obtained by adding a compound in which both the benzene and the salt are soluble (*e.g.*, “hydrohexalin,” derived from methylcyclohexanol).

C. O. HARVEY.

Preparation of stable emulsions of coal dust in oil. A. RIEBECKSCHE MONTANWERKE A.-G. (G.P. 444,420, 20.6.25).—Mono- or poly-hydric phenols, their homologues, derivatives, or mixtures of these containing small quantities of alkali, are used as stabilising agents in the preparation of coal dust-oil emulsions suitable for use as fuel oils. No deposition occurs from such emulsions after being kept for weeks.

A. B. MANNING.

Preparation of aqueous emulsions of paraffins. CHEM. FABR. DR. J. WIERNICK & Co., A.-G. (F.P. 618,918, 13.7.26. Ger., 14.7.25).—Very stable emulsions are obtained by using two emulsifying agents together, in particular vegetable gum and seaweed mucin.

A. B. MANNING.

Dehydration of oil [emulsions]. B. HILDEBRAND (U.S.P. 1,650,514, 22.11.27. Appl., 2.4.25).—An emulsion of oil and water is compressed, expanded, and subjected to forcible impact on a heated surface. It is then passed in a tortuous path through restricted spaces between particles of successive layers of finely-divided material, whereupon on allowing it to settle the oil rises through the water.

H. HOLMES.

Resolution of emulsions or suspensions containing tar or oil. H. W. ROBINSON and D. W. PARKES (B.P. 280,059, 24.12.26).—A solid hydrocarbon melting below 100°, *e.g.*, naphthalene, is added to the slightly acidified emulsion, and the mixture, after heating until the hydrocarbon dissolves, is suddenly cooled. The hydrocarbon is precipitated in the form of fine crystals which agglomerate with the tar or oil and promote separation of the water. The process is applicable to emulsions of water in oil, water-gas tar, or tars containing creosote emulsions.

S. PEXTON.

Process and apparatus for evaporating [hydrocarbon] oils. C. W. STRATFORD (B.P. 280,727, 13.12.26).—See U.S.P. 1,613,298; B., 1927, 274.

Distillation of crude oil from shale. C. A. SPOTZ (B.P. 281,769, 9.9.26).—See U.S.P. 1,601,777; B., 1927, 35.

Filter for liquids (B.P. 280,802). **Supplying liquids in definite proportions** (B.P. 280,010). **Treatment of gases** (B.P. 280,268).—See I. **Water-gas** (B.P. 280,763). **Carbon dioxide** (B.P. 271,852).—See VII. **Artificial asphalt** (B.P. 280,085).—See IX.

III.—ORGANIC INTERMEDIATES.

Catalytic preparation of formaldehyde. B. NEUMANN and P. BILJEVIC (*Z. angew. Chem.*, 1927, 40, 1469—1474).—On thermochemical grounds it is not to be expected that the reversible reaction between carbon monoxide and hydrogen, in which formaldehyde is formed, should yield under equilibrium conditions more than traces of the latter substance within industrial limits of temperature and pressure. On passing carbon monoxide and hydrogen and water vapour at ordinary pressure through an electrically-heated, glazed porcelain tube only traces of formaldehyde were obtained in spite of variations in the composition of the gaseous mixture and the temperature. The presence in the hot tube of silica gel influenced the reaction favourably, 0.525% of the carbon monoxide used being converted at 470° into formaldehyde; cerium oxide and vanadium pentoxide also caused a noticeable improvement in yield, in spite of the fact that the latter readily brings about the decomposition of formaldehyde. Soda-lime did not increase the amount of formaldehyde, though some oxalic acid was formed in this reaction. The gaseous mixture of carbon monoxide and hydrogen was passed through fuming sulphuric acid, and then into the heated tube containing powdered cuprous chloride as catalyst, in the hope of bringing about the intermediate formation of methyl chloride, which would then decompose giving formaldehyde and hydrogen chloride; the best yield obtained under these conditions was, however, only 0.078% of the carbon monoxide used at 270—300°. The mixed gases were passed with chlorine over activated charcoal without perceptible increase in the amount of formaldehyde formed. Carbon monoxide and hydrogen when passed through heated tubes containing cupric chloride gave no formaldehyde but much carbon dioxide; palladium sponge gave only traces of formaldehyde. A mixed nickel-iron-palladium catalyst yielded no formaldehyde, 12—27% CO₂, and 22—37% CH₄ at 255—310°; with iron-cobalt on pumice liquid hydrocarbons were formed. W. J. POWELL.

Preparation of pyrrole. F. F. BLICKE and J. L. POWERS (*Ind. Eng. Chem.*, 1927, 19, 1334—1335).—Ammonium mucate, prepared by adding mucic acid to excess of ammonia solution, is mixed with hot glycerol at 100°, and the mixture heated and distilled for 2—3 hrs. The distillate, consisting of water, ammonium carbonate, and pyrrole, is collected, and the latter separated, dried with solid sodium hydroxide, and redistilled. The yield of pure material on small- and large-scale experiments varies from 40 to 52% of the calculated amount; no advantage was gained by saturating the glycerol with ammonia or by passing the gas through the apparatus during the reaction. F. R. ENNS.

Phenols from wood tar. KLASON and MELLQUIST.—See II. **Explosions with ether.** BRANDT. **Anthranilic esters.** WAGNER.—See XX.

PATENTS.

Manufacture of acetic anhydride. H. DREYFUS (B.P. 280,972, 26.5.26).—Acetic anhydride is obtained by passing acetic acid vapour over a heated mass of one or more phosphoric acids (*e.g.*, ortho-, meta-, or pyrophosphoric acids), at temperatures above the b.p. of the anhydride (preferably 150—400°), and, if desired, under reduced pressure. The anhydride is recovered from the reaction gases by fractional condensation as in B.P. 256,663 (B., 1926, 897). The phosphoric acids may be regenerated by heating. B. FULLMAN.

Concentration of volatile aliphatic acids. HOLZVERKOHLLUNGS-IND. A.-G. (B.P. 271,044, 18.3.27. Ger., 17.5.26).—Dilute aliphatic acids (*e.g.*, acetic acid) are treated with water-combining media, such as sodium hydrogen sulphate or sulphuric acid or both, and distilled so that the vapours come into contact with sodium hydrogen sulphate or salt mixtures containing it. Concentrated acids are obtained; *e.g.*, 10% acetic acid is converted into 80—90% acid. The sodium hydrogen sulphate may be regenerated from the residual liquid, and the organic acid remaining in the latter is recovered by esterification. If the original acid is impure, as in the case of raw pyroligneous acid, the salt (especially sodium hydrogen sulphate) initially added or flowing in during the distillation assists in the separation of tarry or oily matter. B. FULLMAN.

Concentration of raw pyroligneous acid. HOLZVERKOHLLUNGS-IND. A.-G. (B.P. 271,828, 25.3.27. Ger., 25.5.26. Addn. to B.P. 271,044; preceding).—If the acid liquor used as in the prior process contains an alcohol (*e.g.*, raw pyroligneous acid containing methyl alcohol), the presence of drying media during the distillation results in the formation of esters, which are separated as such, the main quantity of acid being separated in concentrated form. If the raw acid be saturated with sodium hydrogen sulphate, the latter serves simultaneously for the removal of tarry matters. B. FULLMAN.

Manufacture of acetaldehyde and acetaldehyde-amine condensation products. RUBBER SERVICE LABORATORIES Co., Assees. of C. N. HAND (B.P. 269,556, 12.4.27. U.S., 12.4.26).—Acetaldehyde, prepared for example by passing acetylene at 25 lb./sq.in. into 25—35% sulphuric acid containing mercurous sulphate at 68—80°, is removed from the gaseous reaction product by combination with a primary amine (aniline, toluidines, xylidines, etc.), preferably in a series of four scrubbing towers, each followed by a condenser and trap for removal of water formed. The acetylene escaping from the last tower is scrubbed and returned to the circuit. C. HOLLINS.

Production of calcium formaldehyde-sulphoxylate. J. Y. JOHNSON. From I. G. FARRENIND. A.-G. (B.P. 281,134, 14.2.27).—A difficultly soluble calcium formaldehyde-sulphoxylate, containing 4 atoms of sulphur to 3 atoms of calcium, is obtained by treating a solution of an alkali formaldehyde-sulphoxylate with calcium chloride (or other soluble calcium salt) and calcium hydroxide. The latter may be replaced by sodium or potassium hydroxide together with a soluble calcium salt, or by calcium or alkali carbonate. Alter-

natively, calcium hydroxide is added alone, the calcium chloride being formed by addition of hydrochloric acid. The same compound is produced by treating mono-calcium formaldehyde-sulphoxylate with calcium hydroxide or carbonate, added as such or formed in the solution.

B. FULLMAN.

Manufacture of aqueous emulsions and suspensions of liquid or solid substances insoluble in water. I. G. FARBENIND. A.-G. (B.P. 252,392, 21.5.26. Ger., 22.5.25).—Certain wetting-out agents, selected as possessing high wetting, dispersing, and emulsifying power, are mixed with insoluble liquids or solids. The agents are non-tanning, aromatic sulphonic acids derived from hydrocarbons carrying as nuclear substituents one or more halogen atoms or nitro-, hydroxyl, or amino-groups, and having in the nucleus or in the amino-group one or more alkyl groups of two or more carbon atoms. Quaternary ammonium salts are excluded. Examples are: diethylmetanilic acid, diamyl- α -naphthylaminesulphonic acids, chloroisopropyl-naphthalene-sulphonic acids, isopropyl-naphthol-sulphonic acids, and their salts.

C. HOLLINS.

Manufacture of iodine-substituted benzonitriles of the phenol ether type. CHEM. FABR. AUF ACTIEN (VORM. E. SCHIERING) (B.P. 275,213, 16.7.27. Ger., 2.8.26).—Iodinated alkoxycyanodiphenyl ethers are prepared by condensing appropriate alkali phenoxides and halogen compounds in presence of copper at 210–240°. 2:6:3'-*Tri-iodo-4'-methoxy-4-cyanodiphenyl ether*, m.p. 154°, is obtained from 3:5-di-iodo-4-hydroxybenzonitrile and 2:4-di-iodoanisole, or from 2-iodo-4-hydroxyanisole, m.p. 92° (prepared from 2-iodo-*p*-anisidine), and 3:4:5-*tri-iodobenzonitrile*, m.p. 196° (prepared from 3:4:5-*tri-iodoaniline*). The products are intermediates for preparation of therapeutically valuable substances allied to thyroxin.

C. HOLLINS.

IV.—DYESTUFFS.

Acceleration of the reduction of indigo by pyridine. A. BINZ and G. PRANGE (Z. angew. Chem., 1927, 40, 1474–1476).—Indigo-carmin, which suffers no reduction with aqueous hydrogen sulphide in the cold in 20 min., is completely reduced in 0.5–1 min. on addition of a small quantity of a base such as sodium hydroxide, ammonia, piperidine, pyridine, collidine, aniline, quinoline, or dimethylaniline (in order of decreasing activity). The reduction of indigotin by hydrogen sulphide takes place in 5 min. with piperidine as solvent, in 10 min. with pyridine, more slowly with methyl alcohol, and very slowly in aqueous solution. Addition of water to pyridine lowers its accelerating power; thus, with a solvent containing 3 pts. of water to 1 pt. of pyridine, no reduction takes place. Reduction of indigo by alkaline hyposulphite, dextrose, formaldehyde-sulphoxylate, or sodium sulphide is also accelerated in presence of pyridine (cf. Binz and Marx, B., 1910, 1200).

W. J. POWELL.

PATENTS.

Manufacture of black and grey vat dyes. BRIT. DYESTUFFS CORP., LTD., A. SHEPHERDSON, and S. THORNLEY (B.P. 280,652, 24.8.26 and 23.6.27).—A sulphonated benzanthrone (e.g., products of sulphonation of benzanthrone, methylbenzanthrone, chlorobenzanthrones, etc.)

is condensed with hydroxylamine or its salts in presence of concentrated sulphuric acid and ferrous sulphate, and the product is fused with alkali, giving a bluish-grey to bluish-black vat dye. The sulphonation and condensation with hydroxylamine may be performed without isolation of the sulphonic acid.

C. HOLLINS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G., ASSEES. OF FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (B.P. 257,618, 26.8.26. Ger., 26.8.25).—Ethers of 3-hydroxybenzanthrone containing no 4-substituent (obtained from the 3-halogen compounds and alkaline alkoxides or aryloxides) are fused with (alcoholic) alkali. 3-*Benzanthranyl methyl ether*, m.p. 172°, gives a violet vat dye, the *phenyl ether*, m.p. 185°, a reddish-violet. Other examples are *bromo-3-benzanthranyl methyl ether* (from dibromobenzanthrone) for bluish-violet, and *phenyl 3-amino-3-benzanthranyl ether* (by nitration of phenyl 3-benzanthranyl ether and reduction) for bluish-grey.

C. HOLLINS.

Manufacture of a solubilised vat dye and dyeing therewith. BRIT. DYESTUFFS CORP., LTD., and A. J. HAILWOOD (B.P. 280,647, 19.8.26).—Improved vatting properties result when the claret dye obtained by alkaline fusion of naphthalimide (B.P. 26,690 of 1913) is dissolved in oleum or chlorosulphonic acid at 50°, cooled, and precipitated by dilution with ice and water. The product disperses easily in warm water and gives a clear vat with alkaline hyposulphite.

C. HOLLINS.

Manufacture of new [acid] dyes of the anthracene series. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 280,637, 17.8.26).—4-Halogeno-1-amino-anthraquinone-2-sulphonic acids are condensed with alkyl- or aralkyl-amines in presence of copper or copper salts. 1-*Amino-4-butylaminoanthraquinone-2-sulphonic acid* gives on wool a clear greenish-blue, the corresponding 4-*benzylamino-derivative* a clear reddish-blue.

C. HOLLINS.

Production of dyes from benzanthrone. BRIT. ALIZARINE CO., LTD., W. H. DAWSON, and P. BEGHIN (B.P. 281,016, 26.8.26).—The non-dyeing by-product obtained in the preparation of dibenzanthrone and *isodibenzanthrone* by alkaline fusion of benzanthrone or its 3-halogen derivatives may be partly converted by alkaline fusion of a different type into further quantities of dibenzanthrone or *isodibenzanthrone*. E.g., the by-product extracted with aniline from an alcoholic potassium hydroxide fusion is heated with aniline and potassium hydroxide to give dibenzanthrone. The by-product from a potassium-aniline fusion gives *isodibenzanthrone* when fused with alcoholic potassium hydroxide.

C. HOLLINS.

Manufacture of dyes, and dyeing of cellulose esters. C. M. BARNARD, and BRIT. ALIZARINE CO., LTD. (B.P. 281,114, and 281,213, 6.1.27).—(A) Azo dyes containing one or more amino-groups and no sulphonic groups are fused with citric acid to give water-soluble dyes for acetate silk: e.g., the dye from reduced *p*-nitrobenzeneazo- β -naphthol (also synthesised from *p*-nitroaniline heated with citric acid, reduced, diazotised, and coupled with β -naphthol) gives orange-brown shades. (B) Indigos and thioindigos containing primary amino-groups (e.g., 7:7'-diaminithioindigo) when fused with

citric acid give water-soluble products suitable for dyeing cellulose esters (cf. B.P. 258,960; B., 1926, 974).

C. HOLLINS.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

State of aggregation of cellulose acetate. H. PRINGS-HEIM, W. KUSENACK, and K. WEINREB (Papier-Fabr., 1927, 25, 785—789).—Primary cellulose acetate may be converted by heating at about 250° in an inert medium such as naphthalene or tetralin into an acetone-soluble, secondary cellulose acetate of lower viscosity without loss of acetic acid (cf. Knoevenagel, B., 1915, 134), but the readiness with which the change takes place varies with different samples. In an examination of the effects of heat and of sodium hydroxide solution on the state of aggregation of cellulose, as determined by viscosity measurement on the sample itself and on the acetate prepared from it, it was found that a sample of cellulose of relative viscosity 5.41 (determined in cuprammonium solution) on drying at 125° yielded a product of viscosity 3.49. The action of 50% sodium hydroxide solution on the same cellulose resulted in a lowering of viscosity to 2.58 after 4 hrs., and on saturating the sample with three times its weight of 16% sodium hydroxide and keeping the product for 3 days the viscosity fell to 1.62. The viscosity of the cellulose acetate (determined in tetrachloroethane) prepared from the dried sample was not reduced, but that of the product from alkali-treated cellulose (4 hrs. with 50% sodium hydroxide) fell from 2.13 to 1.64, and the viscosity of the product from cellulose after 3 days with 16% sodium hydroxide was 1.53. The viscosities of samples of cellulose obtained by hydrolysis of the acetates ran parallel with those of the acetylated products, and the cellulose acetate prepared from alkali-treated cellulose suffered a further diminution in viscosity after heating at 250—255° with naphthalene. The cellulose obtained by hydrolysis from the acetate after this heat-treatment had a viscosity of only 1.24. The lowest viscosity obtained by heat-treatment of cellulose acetate was 1.19, but even this sample was not soluble in methyl alcohol, and, therefore, the cellulose from which it was derived still possessed a higher state of aggregation than glucose anhydride.

W. J. POWELL.

Colloid chemistry of viscose solutions. VII. **Modification of Hottenroth's method for determining "ripeness."** T. MUKOYAMA (Kolloid-Z., 1927, 43, 349—353).—In the above method the ripeness of viscose solutions is determined by measuring the number of c.c. of a 10% ammonium chloride solution (ripeness value) required to coagulate the given solution. The method does not give accurate results, particularly with freshly-made solutions. It is shown that good results are obtained if viscosity measurements are also made. The relation between relative viscosity and ripeness value at 17° for viscose solutions (8% of cellulose, 6.45% of alkali) of varying age is examined. The curves show a sharp maximum in the viscosity especially with the freshly prepared solutions. Increasing alkali content increases the ripeness number.

O. J. WALKER.

Viscose. Conditions of formation of cellulose xanthate. M. NUMA (Kunstseide, 1927, 9, 597—599).—

Combined sulphur in cellulose xanthate (viscose) is determined by treatment with dilute acetic acid and a saturated solution of sodium chloride, oxidising in two stages at a temperature not exceeding 20° the sulphur present by the addition of a solution of sodium hypochlorite containing 5% of available chlorine, and then acidifying the product after 1 hr. with hydrochloric acid, the sulphuric acid formed being determined, after 10 min., in the usual manner. If viscose is prepared with insufficient carbon disulphide, the residual alkali-cellulose hydrolyses to cellulose and free caustic soda. In the preparation of viscose the maximum yield is obtained when the combined sulphur present does not increase with addition of carbon disulphide. Increase of temperature during the ripening of viscose appreciably diminishes the amount of combined sulphur, but not the combined alkali; the former is decreased 65% by ripening at 30° instead of 10°. Sulphiding of alkali-cellulose yields the most satisfactory results when effected at 15°; above 20° a gradual decrease in the amount of combined alkali occurs; and at 50° irreversible coagulation of the viscose commences. Alkaline solutions of viscose (suitable for spinning) retain a high and constant content of combined sulphur during the second to the fifth day of ripening if the temperature does not exceed 30°, but above 30° the combined sulphur decreases considerably so that the solution becomes unstable. The optimum period of ripening is 1—4 days, the viscose solution then having the lowest density, the highest viscosity, and the maximum intensity of colour. The higher the initial viscosity of a viscose solution, the greater the elasticity and other desirable qualities of the resulting cellulose filaments or films. During the ripening of viscose the cellulose (after regeneration) acquires a 70% increase in moisture absorptive capacity, such increase being independent of the period of ripening. The copper number of the cellulose increases slowly during the optimum period of ripening of a viscose solution, after which it increases rapidly. A simple method for determining if a solution of viscose is satisfactorily ripened consists in acidifying it with 20% sulphuric acid; if within the optimum period the precipitated cellulose is fibrous, but beyond this it is powdery. After ripening for 3 days viscose reaches its maximum degree of dispersion.

A. J. HALL.

Formation of oxycellulose and carbon dioxide from cellulose. H. DITZ (Z. angew. Chem., 1927, 40, 1476—1478).—Polemical. Previous work on the liberation of carbon dioxide during the oxidation of cellulose by means of ozone, and by oxygen in ultra-violet light and in sunlight, is discussed.

W. J. POWELL.

Spinning-centrifuge corrosion in artificial silk manufacture. J. EGGERT (Chem.-Ztg., 1927, 51, 961—962).—Deterioration of the aluminium spinning boxes is attributed to insufficient purity of the metal, lack of homogeneity throughout its mass owing to non-uniform cooling after casting (thus producing different degrees of toughness and density in the metal particles), to the formation of bubbles or cavities, or the enclosure of particles of dirt of metallic nature during casting, and to the action of the constituents of the spinning bath. In order to be satisfactorily resistant to chemical re-

agents, aluminium should be not less than 98% pure. Alloying with another metal or even intimate contact with a foreign metal causes a decrease in resisting power, attack occurring especially in the presence of acids (including carbon dioxide), acid salts, or easily dissociated salts. All the inorganic acids used in artificial silk manufacture dissolve aluminium more or less quickly, the action being uniform, however, over the whole surface. Lining the box with pore-free hard rubber is not always satisfactory, since a loose-fitting lining produces the same effect as if the casting contained bubbles or cavities. Alloying aluminium with silicon has no advantages. Rapid destruction is chiefly caused by the formation of electric elements between particles of foreign metals, or the phosphor-bronze bearings, and the aluminium in the presence of acidic substances. The drawn, dense metal is more resistant to chemical reagents than less tough aluminium, and a highly polished surface than a rough one. Impurities in the sulphuric acid are harmful, whilst salts of the alkaline-earth or heavy metals, antimony from the hard lead channels used, too high a salt content of the bath, and especially traces of mercury salts are dangerous.

B. P. RIDGE.

PATENTS.

Treatment of fibres or fibrous materials with aqueous liquids. I. G. FARBERIND A.-G. (B.P. 278,752, 21.5.26. Ger., 22.5.25).—Non-tanning, polynuclear, aromatic sulphonic acids containing one or more substituent halogen atoms or nitro-, amino-, or hydroxyl groups, and carrying nuclear or *N*-alkyl groups of two or more carbon atoms, possess high wetting and/or cleaning power, and are thus good substitutes for soaps. They are not precipitated by acids or calcium salts. Examples are: diamyl- α -naphthylaminesulphonic acids, chloroisopropyl-naphthalenesulphonic acids, isopropyl-naphtholsulphonic acids, and their salts. C. HOLLINS.

Manufacture of artificial textile fibres. BRIT. DYESTUFFS CORP., and A. J. HAILWOOD (B.P. 281,117, 30.11.26).—In the xanthate process for artificial silk from cellulosic materials all or part of the caustic alkali used may be replaced by organic sulphonium hydroxides, $\text{SRR}'\text{R}''\text{OH}$, e.g., trimethylsulphonium hydroxide.

C. HOLLINS.

Treatment of fabrics [containing cellulose esters or ethers]. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 280,989 and 280,990, 27.7.26).—"Slipping," "splitting," and "laddering" of woven or knitted cellulose ester or ether materials, due to lack of cohesion between the threads, may be minimised or prevented, without loading, (A) by impregnating the fabric at 80° with aqueous solutions or dispersions containing at least 8% of one or more of the following substances: (i) higher fatty acids or sulphonated fatty acids or their soluble salts, (ii) sulphaaromatic fatty acids or salts, (iii) resin soaps; (B) by treatment at 80° with an organic solvent for cellulose esters or ethers (e.g., toluene, methylated spirit) or with solutions or dispersions of such solvents of at least 5% strength (e.g., xylene and Turkey-red oil in water). In both cases the impregnated fabric may be left to mature before scouring and finishing.

C. HOLLINS.

Production of sulphite pulp. J. B. BEVERIDGE (U.S.P. 1,649,942, 22.11.27. Appl., 9.6.25).—Wood

particles are digested with a solution containing magnesium bisulphite, an alkali sulphite, and an alkali bisulphite.

H. HOLMES.

Recovery of soda from its solutions [used in treatment of esparto etc.]. W. M. WALLACE and J. MACGREGOR (B.P. 281,035, 13.9.26).—The concentrated, calcined spent liquor is discharged from a furnace on to a travelling grate and delivered into a dissolver of small size fitted with an agitator to break up large pieces and then to a tank communicating both with the dissolver and causticising pots, into which the liquor is delivered when sufficiently concentrated, a continuous circulation of liquor being possible between the dissolver and the tank (cf. B.P. 217,468; B., 1924, 707).

W. G. CAREY.

Manufacture of hollow artificial textile fibres. J. E. G. LAHOUSSE, ASSR. to SOC. FABR. SOIE "RHODIASETA" (U.S.P. 1,652,206, 13.12.27. Appl., 25.10.26. U.K., 11.11.25).—See B.P. 267,187; B., 1927, 361.

[Beating] apparatus for hydrating fibrous pulp for the manufacture of paper and cellulose therefrom. H. JACKSON (U.S.P. 1,652,720, 13.12.27. Appl., 3.4.25. U.K., 10.4.24).—See B.P. 233,501; B., 1925, 496.

Manufacture of cellulose acetate. H. J. MALLABAR (U.S.P. 1,652,573, 13.12.27. Appl., 29.6.25).—See B.P. 258,020; B., 1926, 975.

Spinning of viscose solutions. R. SAJITZ and E. POTT (CHEM. FABR. POTT & Co.), and F. POSPIECH (B.P. 280,608, 17.6.26).—See F.P. 617,600; B., 1927, 963.

Manufacture of substitutes for horn and ivory from viscose. II. EGGERT (U.S.P. 1,652,711, 13.12.27. Appl., 26.3.24. Ger., 29.12.22).—See B.P. 226,071; B., 1925, 126.

Apparatus for drying textile goods in stages. P. SCHIRP (B.P. 269,512, 30.3.27. Ger., 15.4.26).

Drying of paper, board, pulp, etc. R. MARX (B.P. 280,776, 10.3.27).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Mode of origin of colours [in dyeing]. II. Absorption of simple aromatic compounds by cellulose. K. BRASS (Z. angew. Chem., 1927, 40, 1218—1225; cf. B., 1925, 986).—Cellulose of various origin was immersed in dilute aqueous solutions (0.05—0.95%) of different organic compounds at room temperature for 24 hrs., the concentration of the solution being determined before and after immersion. With feebly dissociated phenols a neutral solution was employed, but with pyrogallol it was necessary to use a weakly acid solution. Phenol, pyrocatechol, quinol, methylquinol, and pyrogallol are taken up from their aqueous solutions by both native and reprecipitated cellulose (viscose), the effect being one of dissolution, with a constant partition coefficient between the water and cellulose. The quantity of substance dissolved by the cellulose increases with an increasing number of hydroxyl groups, but is independent of their position and of the introduction of a methyl group. Benzo- and tolu-quinones are not taken up from their aqueous solutions by cellulose.

The influence of the nature of the cellulose surfaces is shown by the increasing quantity of phenols dissolved in passing from cotton to pure cotton cellulose, mercerised and bleached cotton, "copper silk," and viscose. Salicylic and picric acids are adsorbed from their aqueous solutions by cellulose, the order for the different types of cellulose being as before. Acetylcellulose takes up quinol from its aqueous solutions, but the partition coefficient falls with increasing concentration, although quinol is not associated at the concentrations in question.

W. T. K. BRAUNHOLTZ.

Is digallic acid identical with tannin as a mordant for basic dyes? A. GUENTHER (Z. angew. Chem., 1927, 40, 1317—1318; cf. Victorov, B., 1927, 104).—Digallic acid prepared by acetylating gallic acid and treating it with phosphorus pentachloride can be used to mordant cotton with basic dyes, but the colour is not fast to washing and it is not comparable with tannin. Tannin prepared from the synthetic digallic acid is analogous in its behaviour to natural tannin and pentagalloylglucose prepared from gallic acid and dextrose also produces fast dyes on cotton. The author therefore concludes that tannin does not hydrolyse in the dyeing process. Further evidence in support of this is that dextrose cannot be detected in the dyeing bath nor on treatment of tannin with acetic acid, and that only compounds of high mol. wt. act as mordants for cotton.

W. G. CAREY.

PATENTS.

Dyeing [with vat dyes]. J. I. M. JONES, W. KILBY, and STANDFAST DYERS & PRINTERS, LTD. (B.P. 280,983, 28.6.26).—Vat dyes are applied to textile materials in the form of their water-soluble esters (cf. Vaucher and Bader; B., 1924, 864) so that the material is not subjected to the action of strongly alkaline reducing liquors. The material is impregnated with a solution of the water-soluble vat dye and an oxidising agent which remains inactive during steaming, then steamed, and passed through a suitable developing bath. Alternatively, the oxidising agent is omitted from the impregnating liquor and added to the developing bath. *E.g.*, fabric is padded with a solution consisting of 10–50 pts. of Soledon Jade Green, 1 pt. of sodium nitrite, 8 pts. of 6% gum dragon thickening, and 1 pt. of a soluble oil, then steamed for 10 min. in a small Mather-Platt, and the full green shade developed by passage through a 1% solution of sulphuric acid, washing, and soaping.

A. J. HALL.

[Vat] dyeing process. M. E. TICE, Assr. to DUTCHESS BLEACHERY, INC. (U.S.P. 1,652,649, 13.12.27. Appl., 28.2.25).—Material is dyed with vat dyes by successive immersion in concentrated and dilute dye liquors, oxidation of the dyed material between the immersions being avoided.

A. J. HALL.

Dyeing of viscose filaments. S. A. NEIDICH (U.S.P. 1,652,832, 13.12.27. Appl., 3.3.26).—Viscose filaments, immediately after spinning and emergence from the coagulating bath, are drawn singly through a dye-liquor at a rate exceeding 250 yards/min. and then dried under tension, the even distribution of dye liquor within the filaments being obtained by centrifugal force.

A. J. HALL.

Dyeing of acetate silk. I. G. FARBERIND A.-G. (F.P. 619,329, 27.7.26. Ger., 15.8.25).—Acetate silk is dyed with unsulphonated azo dyes derived from diazotised β -hydroxyethyl ethers of aminophenols. Examples are: 4- β -hydroxyethoxyaniline diazotised and coupled with aniline, acetylated (yellow), with 1:7-aminonaphthol (red), with 2:7-aminonaphthol (orange-red); 2- β -hydroxyethoxyaniline with β -naphthol (red), with salicylic acid (yellow); 3-chloro-4- β -hydroxyethoxyaniline with 1-phenyl-3-methyl-5-pyrazolone (yellow); 5-nitro-2- β -hydroxyethoxyaniline with resorcinol (yellow).

C. HOLLINS.

Dyeing of furs, hair, feathers, etc. I. G. FARBERIND A.-G. (F.P. 618,723, 9.7.26. Ger., 10.7.25).—The diamino- β -naphthol (probably 3:8-), obtained from naphthalene-1:2-diazo oxide by dimitration, cautious treatment with sodium sulphide, and subsequent reduction, gives by oxidation in the usual manner for fur bases a greenish-brown on iron mordant, deep brown on chrome, and black-brown on copper.

C. HOLLINS.

Mordanting and colouring of materials made of or containing cellulose derivatives [cellulose esters and ethers]. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 280,698, 30.10.26).—In the mordanting of acetate silk with the assistance of thiocyanates, the use of a quantity of thiocyanate (*e.g.*, of ammonium) in excess of the equivalent of the mordanting metal (iron, chrome, aluminium) improves the absorption of the mordant by the material.

C. HOLLINS.

Manufacture of products [water-soluble esters of vat dyes] for dyeing or printing textile fibres and other materials. DURAND & HUGUENIN S.A., Assocs. of FARBERFABR. FORM. F. BAYER & Co. (B.P. 260,303, 25.10.26. Ger., 26.10.25. Addn. to B.P. 186,057; B., 1924, 252).—Leuco-derivatives of vat dyes are treated with pyrosulphuryl chloride instead of chlorosulphonic acid as described in the chief patent; the presence of a small amount of moisture in the leuco-vat dye is thus permissible, such moisture merely having the effect of converting the pyrosulphuryl chloride into the equally effective chlorosulphonic acid.

A. J. HALL.

Dyeing with solubilised vat dye. (B.P. 280,647).
Dyeing of cellulose esters (B.P. 281,114 and 281,213).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Potentiometric titration of boric acid in the presence of certain inorganic salts. M. G. MELLON and F. R. SWIM (Ind. Eng. Chem., 1927, 19, 1354—1355).—The effect of the chlorides of sodium, lithium, and calcium on the p_H value of a solution of boric acid increases in the order of the salts named, and with the rise in concentration of the individual salts. The fall in p_H value observed on the addition of sodium chloride is relatively small, but 50 pts. of calcium chloride added to 1 pt. of boric acid produces a marked change in the p_H value and also in the character of the potentiometric titration curve with sodium hydroxide solution, a flat portion similar to that obtained in the titration of a strong acid with a strong base developing at the stoichiometric end-point. The use of these salts does not enable

a good end-point to be obtained in the titration of boric acid with sodium hydroxide solution, using phenolphthalein as indicator, and they cannot be regarded as satisfactory substitutes for the polyhydroxy-organic compounds generally used for the purpose.

F. R. ENNOS.

Digallic acid as a reagent for earth acids. W. R. SCHOELLER (Analyst, 1927, 52, 702; cf. Schoeller and Deering, B., 1927, 940).—An acidified solution of sodium tungstate gave with tannin a dark brown precipitate; with oxalonibic acid solution a red colour, and on partial neutralisation a crimson precipitate. With oxalotantallic acid a yellow colour was produced, but precipitation was incomplete even on neutralisation.

D. G. HEWER.

Manufacture of sodium nitrate and hydrochloric acid from sodium chloride and nitric acid (or oxides of nitrogen). V. I. NIKOLAEV (J. Russ. Phys. Chem. Soc., 1927, 59, 685–688).—Pure sodium nitrate is obtained by distilling an aqueous solution of sodium chloride and nitric acid saturated at 45°. The reacting substances are used in such proportions that the free acids present in solution form 38% of the total mixture. On saturating the distillate with sodium chloride a mixture of sodium chloride and sodium nitrate is precipitated. On distilling the filtrate, further quantities of the same mixture are obtained. The separation of sodium nitrate from sodium chloride is based on their different solubilities at 100°. Pure hydrochloric acid and nitric acid containing 0.2% of hydrochloric acid are obtained by treating the distillate with concentrated sulphuric acid.

A. RATCLIFFE.

Manufacture of sodium sulphide. P. P. BUDNIKOV (Chem.-Ztg., 1927, 51, 821–823, 842–844, 862–863).—The manufacture of sodium sulphide from Leblanc "alkali-waste" and sodium sulphate has been superseded by the reduction of sodium sulphate with carbon at 850–1000°. The use of nitre cake is accompanied by loss of sulphur and corrosion of the furnace lining; small amounts can, however, be usefully added to the normal sulphate. Molten sodium sulphate also has a destructive effect on the furnace, and alumina-silica or chrome-magnesite linings are used with smooth non-porous surfaces and in large sections. The amount of sodium sulphate used is $2\frac{1}{2}$ –3 times in excess of the carbon, which is pulverised to 2 mm. size and well mixed with it. Often about 5% of soda is added as a flux and to neutralise free acid in the sulphate; the temperature of fusion is also lowered by addition of potassium sulphate or sulphide (cf. B., 1925, 630). A reverberatory furnace with horizontal hearth is usually employed and 650 kg. of 62–65% sodium sulphide per 24 hrs. is an average output, each charge being 96 kg. of sodium sulphate, 8 kg. of sodium bisulphate, and 48 kg. of anthracite, giving 72 kg. of sodium sulphide. The furnace hearth may be divided into two or three parts by partitions with a charge in each, and fired by producer gas. A continuous furnace with a sloping hearth is also used, but in this case a flux of soda or potassium sulphate is specially advisable to avoid excessive temperature and loss by oxidation; the charge flows direct into a lixiviator fitted with a stirrer. Another modifica-

tion is a furnace in which the charge flows from one sloping hearth to another with less slope. In a continuous furnace the content of sodium sulphide in the melt is less than in the intermittent process and the life of the furnace is shorter, but this is more than counterbalanced by economy of fuel and labour. Shaft furnaces have been tried, but the final product contains carbide. When horizontal rotary furnaces heated by producer gas are used, the melt is extracted with water or weak sulphide liquor in an automatic lixiviator on the counter-current principle. After the suspended matter has settled in steam-heated, conical clarifiers the solution is evaporated until the b.p. is 176° (equivalent to 62% Na₂S) and filled into drums or made into flakes. It may be crystallised with 9 mols. of water from a weaker solution after being decolorised with $\frac{1}{2}$ % of sodium cyanide. Insoluble matter from the manufacture is treated with sulphur dioxide and yields sodium thiosulphate.

W. G. CAREY.

Determination of technical sodium diborate.

M. FRANÇOIS and L. SEGUIN (Ann. Falsif., 1927, 20, 542–545).—In the presence of glycerin borax becomes strongly acid and reacts with sodium hydroxide as follows:— $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O} + 2\text{NaOH} = 4\text{NaBO}_2 + 11\text{H}_2\text{O}$. For its determination 1.91 g. of finely-powdered borax are heated on a water-bath with 25 c.c. of water and 50 c.c. of glycerin until completely dissolved (about $\frac{1}{2}$ hr.). After being cooled the solution is titrated with *N*-sodium hydroxide solution, using phenolphthalein solution as indicator. The number of c.c. of standard alkali used, less 0.05 c.c. (half the volume of the last drop), when multiplied by 10 gives the percentage of Na₂B₄O₇·10H₂O in the sample.

F. R. ENNOS.

Caking of ammonium sulphate. W. G. ADAM (Gas J., 1928, 181, 33–34).—Caking is shown to be caused mainly by small crystals formed by evaporation of moisture and also to depend on size and form of crystal. Determination of the area of contact gives a measure of its caking tendency, and shows that no relationship exists between grading and percentage of voids. Immunity from caking is not guaranteed by production of large crystals, but the percentage of voids gives a true indication of the tendency of ammonium sulphate to cake. As the percentage of voids depends largely on the shape of the crystal, the long needle form is the more desirable.

II. S. GARLICK.

Acid processes for the extraction of alumina.

G. S. TILLEY, R. W. MILLAR, and O. C. RALSTON (U.S. Bur. Mines, Bull. No. 267, 1927, 85 pp.).—Whereas the Bayer process for the extraction of alumina requires bauxite low in silica, methods of acid extraction are not affected by silica content, though a low iron content is desirable. The limited supply of low-silica bauxite renders the acid processes of practical interest. In addition to high-silica bauxite, clays containing 25–35% Al₂O₃ and unsuitable for refractories, leucite, alunite, labradorite, and some shales are available as raw materials. With the exception of leucite and certain clays, they require grinding. Roasting increases the speed of dissolution of alumina, and may decrease that of impurities. The action of sulphuric acid on a variety of clays was investigated. It was found that acid above

70% concentration was less effective, and 60% was preferred. This should not be concentrated above 75% during the heating. Working at increased temperature and pressure did not increase the yield. Felspar and mica are little attacked, and silica, besides being a diluent, hinders dissolution of the alumina. The use of about 90% of the theoretical quantity of acid gives a solution of p_H 3.5, from which basic ferric sulphate is largely precipitated. The product filters better and is easier to handle. Efficient separation depends on the use of a concentration below a given maximum and control of p_H . Sulphurous acid and calcium bisulphite were found to give good yields from raw clay at 180°. Published work on extraction with hydrochloric acid, nitric acid, and acid sulphates is summarised, as are also the methods which have been used or proposed for the precipitation of iron from the solution. None of these is considered satisfactory, and crystallisation of the sulphate is thought the better method of separation. The authors have confirmed Wirth's result that if the ratio of Al_2O_3 to Fe_2O_3 exceeds 7.0:2.3 and all the iron is in the ferrous state, pure aluminium sulphate can be crystallised from neutral solutions. They obtained by slow crystallisation from neutral solutions containing Fe^{++} successive crops of aluminium sulphate crystals of increasing iron content. If calcium carbonate was added to render the ferric iron colloidal while the aluminium remained in solution (p_H 3.5–4.0), aluminium sulphate could be crystallised almost free from iron. The crystallisation of aluminium chloride from saturated hydrochloric acid gives crystals free from iron. It is suggested that the residues from the sulphuric acid extraction of clay should be used for making sodium silicate or silica brick. Aluminium sulphate heated for 4 hrs. at 130° gave a partly dehydrated salt which could be ignited without melting. Loss of sulphur trioxide at a practical rate commenced at 800°. It was much more rapid at 1100°. By using an externally-heated muffle with a controlled current of air, decomposition was effected at 750–850° and a gas containing over 8% of oxides of sulphur obtained. Clay, silica, or carborundum is available as retort material. It is suggested in place of air to use steam or sulphur vapour, the latter yielding pure sulphur dioxide. The alumina produced contained only traces of sulphur. A down-draught sintering machine was also used, but in this case the oxides of sulphur are not recovered.

C. IRWIN.

Preparation of bleaching liquor from liquid chlorine. H. WREDE (Papier-Fabr., 1927, 25, 817–824).—Liquid chlorine has almost entirely replaced bleaching powder in the American paper and cellulose industry. The Mathieson (discontinuous) apparatus is chiefly used for the preparation of bleach liquors. It consists of a concrete tank fitted with a lead coil. Chlorine is evaporated within the coil and at the same time cools the milk of lime in which the gas is being absorbed. The temperature must not exceed 35–40°. A weighed quantity of gas is used for each absorption, while the liquor is continuously circulated by means of a pump. Muller's apparatus is similar. The heat exchange is not really necessary when working to only 4.5% of active chlorine, and its omission renders it possible to

design a simple continuous apparatus. In this, absorption takes place in a hard lead coil fitted at the upper end with a jet through which liquid chlorine evaporates. The milk of lime is circulated in the same direction from a concrete tank, to which fresh lime and water are added by an automatic regulator. The yield of active chlorine on gas used is 90%. Hydrated lime is preferably used, and its content of calcium oxide should be determined by distillation with ammonium chloride. If sodium hypochlorite is required, the use of a tower packed with porcelain rings is recommended. In this case the process can be worked so that free hypochlorous acid is produced thus: $Na_2CO_3 + 2Cl_2 + H_2O = 2HClO + 2NaCl + CO_2$, and sufficient soda is added after use to neutralise the hydrochloric acid left. The tower apparatus may also be fed with milk of lime. The transport of chlorine in cylinders, drums, and tank wagons is described.

C. IRWIN.

Composition of bleaching powder. I. E. A. O'CONNOR (J.C.S., 1927, 2700–2710).—The equilibrium composition of a bleaching powder giving any particular ultimate analysis could be determined if data for equilibria in the quaternary system $Ca(ClO)_2$ – $CaCl_2$ – CaO – H_2O were available. As a preliminary investigation the three ternary systems in which water is a component are studied at 0°. In the system chloride–oxide–water, $CaCl_2 \cdot 3CaO \cdot 16H_2O$ was the only basic salt obtained. No double salt was formed by calcium hypochlorite and calcium chloride. The correct composition of the hydrate of the hypochlorite is shown to be $Ca(ClO)_2 \cdot 3H_2O$ (cf. Orton and Jones, B., 1909, 307), and its solubility in water at 0° is 21.8%. In the system $Ca(ClO)_2$ – CaO – H_2O , the two basic salts $Ca(ClO)_2 \cdot 2CaO \cdot 2H_2O$ and $Ca(ClO)_2 \cdot 3CaO \cdot 3H_2O$ were found. The composition of bleaching powder is discussed and the opinion expressed that it is a mixture of calcium hydroxide with partly hydrated calcium chloride and hypochlorite. The preparation of nearly pure calcium hypochlorite by a modification of the Griesheim-Elektron process is described.

O. J. WALKER.

Production of salts of noble metals for electroplating. F. CHEMNITZ (Chem.-Ztg., 1927, 51, 823).—Potassium aurocyanide is made by dissolving gold foil in aqua regia, precipitating with ammonia solution, filtering, washing the fulminating gold with warm water until free from chlorine, and dissolving it while still moist in a slight excess of potassium cyanide solution. The double salt is crystallised after evaporation and dried. Potassium argentocyanide is made by precipitating silver nitrate solution at 70° with potassium cyanide (excess being avoided), washing the silver cyanide by decantation until free from nitrate, and dissolving the precipitate in potassium cyanide solution. Animal charcoal is added, and the solution is filtered, evaporated, and crystallised, the crystals being washed with alcohol and dried at 60°.

W. G. CAREY.

Recommended specifications for analytical reagent chemicals. W. D. COLLINS, H. V. FARR, J. ROSIN, G. C. SPENCER, and E. WICHES (Ind. Eng. Chem., 1927, 19, 1369–1372).—Specifications and test methods recommended by a Committee of the American Chemical Society for the following reagents are published: molyb-

dic acid 85%, molybdic anhydride, phosphoric acid, arsenic trioxide, barium nitrate, carbon disulphide, carbon tetrachloride, potassium dihydrogen phosphate, sodium nitrate, sodium peroxide, disodium hydrogen phosphate, sodium sulphide, and stannous chloride.

C. IRWIN.

Importance of carbon monoxide as a reducing agent in the manufacture of hydrogen by the contact process. A. LOSCHIKAREV (J. Oil Fat Ind. Russ., 1926, [6], 22—25; Chem. Zentr., 1927, I, 2936—7).—In the contact process for the manufacture of hydrogen for use in fat-hardening, the reduction of the iron oxide is brought about more readily by the carbon monoxide than by the hydrogen of the water-gas used, reduction being more rapid the higher the carbon monoxide content of the water-gas. It is recommended therefore that the carbon monoxide content be increased either by returning the gas, after its use for reduction, to the producer, or by addition of oxygen to the air supply of the latter. The fuel used should be as free as possible from sulphur, which otherwise takes up part of the available oxygen. Results are tabulated showing the composition of the water-gas before and after reduction, and illustrating the relatively higher consumption of carbon monoxide than hydrogen. A. B. MANNING.

Production of uniform stains in the Gutzeit test for arsenic. J. R. STUBBS (Analyst, 1927, 52, 699—700).—To ensure that all evolved gas in the Gutzeit arsenic test passes through that portion only of the mercuric chloride test paper covering the end of the glass tube, the tube, with ground end, is inserted into a cork with the end flush with the surface, the test paper placed over the surface, and this cork, together with another similarly bored, inserted into a glass collar, so that the paper is firmly held against the end of the tube. D. G. HEWER.

Method of applying the Gutzeit test for arsenic. C. H. CRIBB (Analyst, 1927, 52, 701).—The discs of mercuric chloride paper are gummed to the upper ground glass surfaces of two tubes of external diameter not less than 11 mm. and bore of 5 mm. These tubes are arranged one over the other, so that all gas evolved must pass through both discs. With 0.01 mg. of arsenic a very faint stain appears on the lower disc, and this becomes bright red-brown on treatment with hydrochloric acid. If more than 0.02 mg. is present the upper disc is also stained. As the test is based on a comparison with the stain produced by 0.01 mg. of arsenic, the quantity of material used should be adjusted accordingly, and the total volume of liquid plus zinc in the 3 oz. generating bottle should not exceed 60 c.c.

D. G. HEWER.

Spring clip for Gutzeit tube. J. WHITE (Analyst, 1927, 52, 700—701).—To ensure good contact between the test paper and Gutzeit tube, a clip may be used which consists of a metal cylinder to the top of which is fixed a metal plate; the cylinder is cemented to the tube so that the plate is about 10 mm. from the top. A movable metal plate with a cylindrical base is connected with a bakelite plate by two metal rods which pass through the first fixed plate. The bakelite plate is held in position by a spring which can be moved up and down to allow insertion of the test paper. The tube,

with a hole about 2 mm. diameter and 10 mm. from the bottom, may be fitted into a wide-mouthed bottle about 120 c.c. capacity.

D. G. HEWER.

Formation of hydrogen sulphide by the natural reduction of sulphates. L. ELION (Ind. Eng. Chem., 1927, 19, 1368).—The reduction of sulphates by bacterial action with evolution of hydrogen sulphide is associated with the oxidation of organic matter which supplies the necessary energy. The number of bacteria known to possess the capacity to reduce sulphates is very limited. They are all non-sporogenic and strictly anaerobic. C. IRWIN.

Composition of ozone and the time-energy factor in ozone formation. P. C. SAUNDERS and A. SILVERMAN (Ind. Eng. Chem., 1927, 19, 1316—1321).—An all-glass apparatus is described in which purified electrolytic oxygen was ozonised, the volume contraction measured, and the resultant gas treated with potassium iodide. Concentrated sulphuric acid in which ozone is insoluble was used as the manometer liquid. The electric discharge was obtained from a transformer working on town's supply. Good agreement was obtained between the volume contraction and the results of the iodide determination, giving no reason to doubt the accuracy of the formula O_3 . Time curves obtained show that equilibrium is reached in a few minutes. For a given spark gap, the ozone concentration obtainable decreased with increasing voltage. The more rapid yield at higher voltages, however, renders these advantageous under commercial conditions, when equilibrium is never reached. For voltages giving the true brush discharge, the yield per coulomb was independent of the voltage. The yield varied inversely with the width of the gap. The maximum concentration of ozone obtained was 16.5%. C. IRWIN.

Manufacture of sulphur monochloride. E. TERLINCK (Chem.-Ztg., 1927, 51, 861—862).—The formation of sulphur monochloride when chlorine is passed into molten sulphur is accompanied by the production of considerable amounts of higher sulphur chlorides, which dissolve in the monochloride, but if the mixed chlorides are heated for some time under a reflux condenser the vapour ultimately assumes the b.p. of sulphur monochloride (137.7°). The reaction therefore to produce only the monochloride is dependent largely on the temperature, and is best carried out in a solution of sulphur in boiling sulphur chloride. An iron vessel is fitted with an iron reflux column and a condenser, and is charged with sulphur and sulphur chloride, which is then heated to boiling. Dry chlorine is passed rapidly into the solution, and when the sulphur is almost completely used up about two thirds of the mixture is distilled and the resulting sulphur monochloride collected, more sulphur then being added and the process repeated. The distillation may proceed while chlorine is still being absorbed, and the amount of chlorine added may be ascertained by the decrease in weight of the chlorine cylinder or by passing the gas through a gas-meter.

W. G. CAREY.

Chromate solutions as colour standards. JØRGENSEN.—See II. **Determination of iodine.** WILKE-DÖRFURT.—See X. **Chemical heating equipment.** ANGEL.—See XI.

PATENTS.

Manufacture of nitric acid. A. HOUGH, Assr. to DURIRON Co., Inc. (U.S.P. 1,649,901, 22.11.27. Appl., 27.3.25).—A mixture of sulphuric acid and a suitable nitrate is heated at distilling temperature in an elongated still having a longitudinal series of rotating agitator discs therein, certain of which are provided with means to elevate the liquid mass when they are partially submerged. By this means the mass is constantly agitated and portions are presented in separate films, thus permitting free escape of acid at a comparatively low temperature. W. G. CAREY.

Extraction and drying of inorganic jellies [gels]. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 279,941, 4.8.26).—Inorganic jellies, *e.g.*, silicic acid gel, are caused to set in plates or sheets in open frames containing lattice work or partitions to support the jelly slabs, the operation being conducted in a rectangular vessel containing, alternately, frames and plates. When the jelly has set the plates are withdrawn and the jelly is washed with a continuous flow of a suitable liquid, and dried in the frames. W. G. CAREY.

Preparation of solutions of hydrofluosilicic acid. W. MÖLLER and W. KRETH (B.P. 263,779, 7.12.26. Ger., 23.12.25).—Silicon fluoride is decomposed by water in an apparatus similar to that used for gas-washing consisting of a horizontal closed tube with a rotating spindle fitted with radially disposed discs or blades dipping partially into the liquid intended to receive the hydrofluosilicic acid. The silicon fluoride is decomposed by the thin aqueous coating on the blades, separated silicic acid is washed off at every revolution, and is removed by suitable means; the reaction surface is kept cool by the liquid. W. G. CAREY.

Production of volatile inorganic acids. II. FRISCHER (B.P. 271,881, 25.5.27. Ger., 27.5.26).—Nitrates, chlorides, etc. are decomposed to the corresponding acids by intermittent or continuous treatment of the finely-divided salt, under reduced pressure and at 200–350°, in a horizontal cylindrical vessel heated internally or externally, with the theoretical amount of sulphuric acid and with the addition of 5–10% of the acid to be produced. It is preferable to mix a part only of the salt and acid to be produced, and then add the remainder of the salt with the calculated amount of the decomposing acid to produce the normal salt. W. G. CAREY.

Production of hydrated sodium carbonate. C. SUNDBSTROM and G. N. TERZIEV, Assrs. to SOLVAY PROCESS Co. (U.S.P. 1,650,244, 22.11.27. Appl., 7.10.26).—A mixture of sodium carbonate and water saturated with respect to the monohydrate is maintained within the temperature range of stability of the monohydrate. W. G. CAREY.

Production of sodium hydrosulphide and valuable by-products. E. B. ALVORD, Assr. to GRASSELLI CHEMICAL Co. (U.S.P. 1,650,106, 22.11.27. Appl., 8.2.26).—A solution of barium sulphide is treated with carbon dioxide up to about one half of the chemical equivalent of the barium present, depending on the amount of sodium sulphide desired in the product,

and the resulting barium carbonate is separated from the solution. W. G. CAREY.

Production of alkali hydride. DEUTSCHE GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 276,313, 15.7.27. Ger., 23.8.26).—An alkali metal is fed gradually into the upper end of a rotary tube-furnace containing a diluent for the alkali metal, *e.g.*, an alkali hydride, and hydrogen, heated above 200°, is introduced in counter-flow to the metal, the requisite heat being wholly or partially introduced by the reaction gas. W. G. CAREY.

Production of ferrocyanides from crude calcium cyanides. J. M. RUGH, Assr. to AMER. CYANAMID Co. (U.S.P. 1,650,390, 22.11.27. Appl., 30.10.23).—Sufficient crude calcium cyanide is mixed with a solution of ferrous sulphate to react with all the sulphate present, and rather less than the amount of sodium carbonate required to convert all the resulting calcium ferrocyanide into the sodium salt is added, the solution is filtered, the remaining sodium carbonate is added, and the solution of sodium ferrocyanide is again filtered. W. G. CAREY.

Production of metal hydroxides free from iron, by means of basic substances, from metal salt solutions containing iron. O. LEDERER, W. STANCAK, and H. KASSLER (B.P. 267,491, 28.2.27. Czechoslov., 9.3.26).—The basic substance (alkali hydroxide, alkali carbonate, ammonia, lime, etc.) is added gradually to the solution in the presence of excess of hydrogen sulphide or a suitable sulphide, which acts as a "reaction buffer," a slightly acid reaction being maintained throughout. The precipitated basic salt is then separated and decomposed into acid and hydroxide by hydrolysis under pressure. W. G. CAREY.

Production of alumina from aluminium nitrate. B. F. HALVORSEN, H. J. FALCK, and Ø. RAVNER, Assrs. to NORSK HYDROELEKTRISK KVALESTOFAKTIESELSKAB (U.S.P. 1,652,119, 6.12.27. Appl., 28.5.24. Norw., 13.6.23).—Aluminium nitrate is fused, converted into spray, and passed into a chamber filled with hot gases (*e.g.*, air at 1000°). W. G. CAREY.

Treatment of titaniferous material. TITAN Co. A./S. (B.P. 275,579, 2.7.27. Norw., 3.8.26).—Ores containing titanium are rendered soluble by acid treatment with the addition, before or during the process, of a reducing agent containing sulphur. Such agent is preferably a metallic sulphide heated and rapidly cooled by immersion in water, and may be titaniferous material heated in sulphur vapour or with a sulphide, *e.g.*, iron pyrites. All the material may be sulphurised by heating in sulphur vapour, and hydrogen sulphide may be introduced during the dissolution process. W. G. CAREY.

Regeneration of catalysts. H. G. C. FAIRWEATHER. From SELDEN Co. (B.P. 280,712, 26.11.26).—Contact masses (particularly vanadium compounds on a polysilicate carrier having a zeolite-like structure), which have been used for oxidations (*e.g.*, naphthalene to phthalic anhydride), are revived by treatment at 300–500° with halogens, non-metal halides, acids, or acid anhydrides, together with oxygen or air; or, after treatment at 300–500° with oxygen, the mass is stirred

with hydrogen peroxide solution which is then removed by evaporation; or the mass is heated slowly to 300° in a steam of ozonised air. The hydrogen peroxide method is especially suitable for iron vanadate catalysts. Amongst the reagents mentioned are: sulphur trioxide, hydrogen halides, ammonium halides, sulphur chloride, thionyl chloride, sulphuryl chloride, phosgene, nitrosyl chloride, ethylene dichloride and dibromide, nitrogen oxides, and acyl chlorides. Addition of steam may be advantageous.

C. HOLLINS.

Simultaneous production of a gas of the nature of water-gas, phosphoric acid, and an aluminium silicate slag having the same composition as a cement. E. URBAIN (B.P. 280,763, 24.2.27. Fr., 20.1.27).—The Wöhler process for the manufacture of phosphorus by the reaction of silica with tricalcium phosphate in the presence of carbon is modified by replacing a portion of the silica by alumina, or alumina and clay, to obtain in the slag 2 pts. of calcium aluminate to 1 pt. of calcium silicate, thus forming a fusible cement slag. Water vapour and a halogen acid are added to the resulting gas containing phosphorus and carbon monoxide, and the phosphorus is oxidised to metaphosphoric acid by passing the gas through amorphous carbon; the hydrogen formed mixes with the carbon monoxide to form a water-gas mixture. W. G. CAREY.

Extraction of carbon dioxide from gaseous mixtures. I. G. FARBEINID. A.-G. (B.P. 271,852, 9.5.27. Ger., 28.5.26).—Gaseous mixtures to be used in the synthetic manufacture of ammonia are scrubbed in a succession of towers filled with Raschig rings with aqueous ammonia circulated by pumps and cooled between each tower. The gases are conducted preferably in counter-flow to the ammonia, and a portion of the circulated liquid is continuously transferred to the preceding tower (considered in relation to the direction of the gas), while ammonia is continuously admitted into the first tower and water or neutral or acid scrubbing solution into the last, and ammonium carbonate solution is continuously withdrawn from the tower (usually the second) into which the gases are first admitted. Any residual traces of ammonia in the purified gases are removed by scrubbing with sulphuric acid in a tower.

W. G. CAREY.

Recovery of sulphur from solutions of ammonium polysulphides. I. G. FARBEINID A.-G. (B.P. 269,516, 12.4.27. Ger., 16.4.26).—The liquid is treated in a column still filled with porcelain balls or Raschig rings with a counter-current of direct steam at such a pressure (2–3 atm.) that sulphur separates at a temperature above its m.p., flows to the lowest part of the column (preferably widened), and is collected continuously or intermittently. The waste solution freed from polysulphides is used for preheating the liquid.

W. G. CAREY.

Purification of sulphur containing bitumen. W. BLYTHE & Co., LTD., W. H. BENTLEY, and B. CATLOW (B.P. 281,133, 12.2.27).—Bituminous sulphur is distilled with 10% of a finely-divided, inert, absorbent or adsorbent solid which is non-volatile at the distillation temperature, e.g., calcium sulphate, bone ash, active carbon, silica gel, or kieselguhr.

W. G. CAREY.

[Preparation of] fluorine. P. M. A. LEBEAU and A. A. L. J. DAMIENS (F.P. 619,605, 7.12.25).—An acid fluoride of an alkali metal or a mixture of such compounds, which fuses below 150°, is electrolysed.

F. G. CROSSE.

Synthesising oxides of nitrogen. C. J. GOODWIN, ASSR. to MATHIESON ALKALI WORKS (U.S.P. 1,652,781, 13.12.27. Appl., 4.9.23. U.K., 5.9.22).—See B.P. 205,288; B., 1923, 1222 A.

Recovery of soda (B.P. 281,035).—See V. **Electrolytic caustic soda (B.P. 281,184).**—See XI. **Potassium solutions from distillers' mash (B.P. 268,790).**—See XVIII.

VIII.—GLASS; CERAMICS.

Determination of boric acid in glass. A. A. MATSCHIGIN and T. KORZUCHINA (J. Russ. Phys. Chem. Soc., 1927, 59, 573–578).—The method is based on the precipitation of impurities, e.g., silicic acid and oxides of aluminium, iron, calcium, magnesium, lead, zinc, which hinder the titration of boric acid, by boiling with barium carbonate. The boric acid is thus converted into barium metaborate, which is dissolved in an excess of barium chloride, the excess of carbonate is removed by boiling with 2*N*-hydrochloric acid, the solution is neutralised with 0.1*N*-barium hydroxide using methyl orange as indicator, and is then titrated with barium hydroxide in presence of mannitol and phenolphthalein.

A. RATCLIFFE.

Slagging of refractory materials [fireclay]. H. SALMANG (Stahl u. Eisen, 1927, 47, 1816–1820). The action of the various constituents of metallurgical slags on crucibles made of a high-grade fireclay containing 56.52% SiO₂, 40.79% (Al₂O₃+TiO₂), and 2.42% Fe₂O₃ has been examined at temperatures up to 1600°. Ferrous and manganous oxides and the fluid basic silicates derived from them are extremely corrosive towards fireclay above 1100°, whilst lime and magnesia have a rapid slagging action above 1500°. The rate of corrosion decreases with increasing acidity of the slag, and viscous slags are much less reactive than the more fluid slags; thus orthoclase feldspar has practically no action on the clay at 1600°. Calcium phosphate dissolves fireclay almost as rapidly as lime does at 1500°.

A. R. POWELL.

Swelling of bentonite and its control. C. W. DAVIS (Ind. Eng. Chem., 1927, 19, 1350–1352).—In wet-drilling for oil difficulty occurs if bentonite is encountered owing to this material having the property of swelling into a sticky paste when in contact with water. This swelling was studied by measuring the increase in apparent volume of a given quantity of the substance placed with water or other solution in a test tube. The swelling, which may be to seven times the original apparent volume, was greatly reduced by the addition of each of a large number of salts to the water, being least with saturated solutions. Addition of acid or alkali had only slight effect between *p_H* 4 and 9. A rise in temperature caused slightly increased swelling. It is concluded that the most satisfactory procedure in many cases is to add as much common salt as possible to the drilling water. Some bentonites become soft in

saturated sodium chloride solution; in such cases some other suitable addition may be found. C. IRWIN.

Effect of the addition of sodium carbonate and silicate on the casting properties of clay slip. F. E. BROWN and C. F. LOI (Proc. Iowa Acad. Sci., 1926, 33, 170).—The plasticity of some clay slips is increased by the addition of small quantities of a mixture of sodium carbonate and silicate. CHEMICAL ABSTRACTS.

Silica retort settings. HAY, also EDWARDS.—See II. PATENTS.

Glass composition. W. C. TAYLOR, Assr. to CORNING GLASS WORKS (U.S.P. 1,652,259, 13.12.27. Appl. 8.4.22).—Translucent glass is prepared from a batch which contains a relatively high proportion of silica, at least 3% of boric oxide, and a little alumina only, and is free from substantial quantities of lead. The opacifying agent is a sulphate, melting being so performed that there is no loss of the sulphur content by volatilisation.

A. COUSEN.

Production of glass resistant to cracking. F. EISENLOFFEL (G.P. 444,749, 29.8.24).—Glass suitable, e.g., for the manufacture of artificial gems contains 5–20% BeO, at least 10% Al₂O₃, not more than 10% of alkalis, and at least 33% SiO₂. L. A. COLES.

Tunnel kilns. WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 280,044, 3.12.26).—A recuperative, annular muffle kiln through which material is moved on a circular track and with a zone of open heating is provided with ducts connecting the cooler parts of the kiln on each side of the zone of open heating with the main flue, to minimise convection currents, facilitate the admission of cooling air, and assist in the extraction of water from on-coming material before it reaches the open-heating zone. A moving platform has transverse, vertical walls spaced at suitable intervals further to minimise convection eddies, and a movable adjustable jamb is provided to make a tight joint with the transverse wall of the kiln. The combustion gases are completely burnt by causing them to impinge upon the sides of the moving platform, and radial slots, opening into an annular central channel, supply radiant heat from the bottom to the material on the platform. To ensure even heating in the open-firing zone, the inlets for the combustion gases and the outlets to the flues are arranged at the bottom of the side walls. W. G. CAREY.

Apparatus for feeding molten glass. BRIT. HARTFORD-FAIRMONT SYNDICATE, LTD., and T. WARDLEY (B.P. 280,611, 18.6.26).

Tunnel kiln. W. L. HANLEY, JUN. (U.S.P. 1,652,570, 13.12.27. Appl. 6.10.23. Renewed 11.3.27).—See B.P. 249,265; B., 1926, 441.

IX.—BUILDING MATERIALS.

Slag, coke breeze, and clinker as aggregates. F. M. LEA and F. L. BRADY (Dept. Sci. Ind. Res., Building Res., Spec. Rept. No. 10, 1927, 22 pp.).—Blast-furnace slag for use as aggregate must not be very acid nor strongly basic, an average amount of silica in an acid slag being 40%, and of lime in a basic slag being 43%. Acid slags are glassy in condition and there is therefore a liability to lack of adhesion between aggregate and

cement, whereas basic slags are more crystalline and form a good key; they also react slightly with the water in the cement. This hydration and consequent expansion is a danger if much fine material is allowed to remain; it is therefore better to reject particles, after crushing the slag, from sand-fineness downwards. Some basic slags disintegrate on cooling, especially those with a high lime content, but a slag which does not begin to powder within 14 days will remain stable indefinitely, except perhaps when exposed to fire; for the same reason only normal air-cooled slag should be used, and it should be well matured. Sulphur is present in fresh slag as sulphide, but is oxidised as the slag weathers; if the sulphide content is not above 1.5–2% and the slag is compact, no deleterious effect is to be anticipated. Coke breeze must be free from coal, shale, and dirt, and should have a low sulphide content. Gas-works coke is usually softer than metallurgical coke, and may contain a higher proportion of sulphur. Concrete made from coke breeze aggregate is weak mechanically and unsuited for outside use and in wet places, but it yields a light product suitable for internal walls, whilst coke breeze is usefully employed with sand as fine aggregate. Clinker for use as aggregate must be free from coal etc., and domestic ashes should never be used. It should be partially sintered, and careful selection is necessary owing to its variable composition and possibility of lines of fissure. Clinker should not contain more than 1% of calcium sulphate and a smaller content of sulphide, with not more than 0.2% of easily soluble sulphates. As in the case of slag it is advisable to sieve out the very fine particles, sand being used to make up the fine aggregate. Clinker makes a stronger concrete than breeze, and has low heat conductivity and good fire-resistant properties.

W. G. CAREY.

Long-time tests of concrete using various coarse aggregates. P. J. FREEMAN (Eng. News-Rec., 1927, 99, 879–880).—Comparative tests of specimens of concrete prepared from slag, limestone, granite, trap-rock, and gravel were made at the end of various periods from 14 days up to 10 years. All specimens were stored in damp sand for 35 days and subsequently in dry warm air until the end of the 5-year period. After 5 years specimens were placed out-of-doors. All concrete, regardless of the type of coarse aggregate, increased in strength when stored in dry air up to the age of 1 year and after that time decreased in strength so long as air-storage continued. Strength was regained during storage out-of-doors, and at the end of 10 years exceeded that at the end of 1 year. The results indicated that all the aggregates were capable of producing concrete having strength considerably beyond the requirements of commercial practice. W. T. LOCKETT.

Shrinkage effect of calcium chloride in concrete. A. S. LEVENS (Eng. News-Rec., 1927, 99, 912).—The increase of strength of concrete by the addition of 2% of calcium chloride (cf. B., 1926, 825) is accompanied by a shrinkage 85% greater than in the case of plain concrete. The maximum shrinkage in concrete results from the addition of 4% of calcium chloride to 1:2:4 concrete, being 150% greater than the plain concrete after 21 days. Although the addition of small quantities may not be objectionable for some work, the use

of calcium chloride is not advisable where shrinkage is an important factor. W. G. CAREY.

Heat changes during setting of alumina and Portland cements. A. A. JAKKULA (Eng. News-Rec., 1927, 99, 955—956).—Specimen cylinders of the two types of cement were allowed to set in an insulated box and the temperature changes recorded. The total heat developed was also determined by setting within a calorimeter. Alumina cement gave a very sudden temperature rise to above 100°, the time of its occurrence depending on the initial temperature. The rise with Portland cement was much more gradual, and the total heat developed was less than half that evolved by a similar alumina cement. C. IRWIN.

Wood preservation. V. Weathering tests on treated wood. L. P. CURTIN, B. L. KLINE, and W. THORDARSON (Ind. Eng. Chem., 1927, 19, 1340—1343).—Wood treated with various preservatives was cut into small pieces and exposed in trays to the action of wind, rain, and sun. It was analysed for loss of preservative after 4 and 10 months' exposure. The samples were further tested for toxicity with 3 different types of culture. Zinc meta-arsenite and copper aceto-arsenite showed losses of nil and 9%, respectively, and the samples were fully resistant to all cultures. Samples treated with barium carbonate, zinc chloride, and sodium fluoride showed very high losses of the preservative and impaired resistance to the cultures. With creosote the loss was 27—48%, and the wood was of reduced toxicity though not actually attacked. Samples treated with petroleum oil showed a loss of 8—19%, but were not resistant to fungus even before weathering. The loss in the case of oil-treated samples is merely a function of the vapour pressure. (Cf. B., 1927, 938.) C. IRWIN.

PATENTS.

Rotary [cement] kiln and cooler. J. S. FASTING, Assr. to F. L. SMIDTH & Co. (U.S.P. 1,652,168, 13.12.27. Appl., 7.12.25).—A rotary kiln with an open end is combined in front with the protective hood of a relatively fixed burner. Within this hood is a kiln-closing hood mounted so as to be movable towards and from the end of the kiln, and, supported on pivots by the protective hood, are beams the inclination of which can be varied. M. E. NOTTAGE.

Calcining cement, lime, etc. in a shaft kiln. A. ANDREAS (G.P. 445,014, 5.8.24).—The finely-divided material, alone or mixed with coal dust, is charged tangentially through tubular openings into the upper part of a shaft furnace in which it is brought in contact with a counter-current of hot gases. L. A. COLES.

Production of coloured cement or cement mixtures. H. E. DOUGHTY (B.P. 280,813, 7.6.27).—The components of "plain cement," cement plaster, gypsum, etc. are ground together with colouring matter in a mill adapted to reduce them to an extremely fine state, the fine particles being drawn off by air-flotation under a vacuum. Sodium silicate or other waterproofing powder is added. W. G. CAREY.

Treatment of hydraulic cement. A. KHOUVINE (F.P. 619,346, 28.7.26. Belg., 25.7.25).—The setting time and hardness of the product are regulated by

adding rare earths, a compound of an element of the fourth group of the periodic system, *e.g.*, 0.3% of titanite oxide, and, if desired, other material, such as calcium sulphate, to lime or cement. L. A. COLES.

Porous material containing cement. BUBBLE-STONE Co. (F.P. 606,042, 27.10.25).—Tiles or sheets of material suitable for use as insulators against heat and sound are prepared from mixtures containing colloidal solutions worked up to a froth with cement, gypsum, etc. The colloidal solutions are prepared, *e.g.*, by vigorously stirring mixtures of solutions in alcohol or acetone of resins, cellulose esters, phenol-formaldehyde condensation products, rubber, etc. with solutions containing formaldehyde and such substances as glue, casein, dextrin, starch, etc. Frothing can be improved by the addition of saponin, soap, etc. L. A. COLES.

Waterproofing materials and concrete articles treated therewith. E. DOGGETT (B.P. 281,022, 27.8.26).—Concrete articles are treated with a composition of 1 pt. by wt. of boiled shellac dissolved in alcohol, acetic acid, or caustic alkali and 8 pts. by wt. of alum boiled with water to a thick liquid, the ingredients being mixed to form a thin paste, and sufficient water is added to carry the mixture into the pores of the concrete. W. G. CAREY.

Treatment of Kentucky and similar rock asphalts in the making of paving material. S. S. SADTLER, Assr. to AMESITE ASPHALT Co. OF AMERICA (U.S.P. 1,650,047, 22.11.27. Appl., 3.3.26).—Sufficient powdered gilsonite is added to rock asphalt reduced to the size of sand to increase the asphalt content to about 9%, the mixture is heated to about 130° and constantly stirred, then allowed to cool while a small quantity of a solvent of asphalt is stirred in to replace any volatile bituminous substances lost during heating. W. G. CAREY.

Preparation of artificial asphalt material from the natural constituents of mineral asphalt. S. G. S. DICKER, From N. V. NEDERLANDSCHE BIMS-CEMENT- EN ASPHALTIND. (B.P. 280,085, 7.3.27).—Limestone or other porous earth is impregnated with 9—12% of bitumen dissolved in a suitable solvent, at a pressure of 3—5 atm. and at about 80°. S. PEXTON.

Materials for use in making or repairing road and like surfaces. A. C. BROWN and J. T. HINES (B.P. 280,011, 11.10.26).—A colloidal dispersion of resin oil in a volatile mineral oil, *e.g.*, kerosene or paraffin, is made in a colloid mill with a suitable dispersing agent, *e.g.*, oleic acid, and crushed stone or other aggregate is treated therewith, an asphalt compound or melted hard bitumen and suitable filling material also being added to the mixture. W. G. CAREY.

Production of objects of artificial stone. F. TRACHSEL, J. WIEDMER, and P. ZIGERLI (B.P. 280,104, 2.5.27).—Organic or asbestos fibres are treated with water-glass solution previously treated with turpentine, a precipitant, *e.g.*, milk of lime, is added to precipitate from the water-glass an insoluble silicate, and finally a binder, *e.g.*, Portland cement, is introduced. W. G. CAREY.

[Portable, continuous-burning] lime kiln. T. DAND (B.P. 281,460, 10.11.26).

Concrete mixers etc. CHITTENDEN & SIMMONS, LTD., and W. B. GUNNELL (B.P. 281,839, 18.1.27).

Slag for cement (B.P. 280,763).—See VII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Heredity of [iron] castings. A. LE THOMAS (Compt. rend., 1927, **185**, 1595—1597).—An attempt is made to explain the different physical properties of iron castings which have the same composition but are of different origin. Thus a grey casting of pearlitic structure and containing 3.25% C was melted, a portion cast in warm sand, and the remainder in a small cooled shell. The graphitic and tempered products thus obtained were then melted under identical conditions and cast into bars in cold sand. Their physical and micrographical properties and chemical analyses, however, then showed only insignificant differences (cf. following abstract). J. GRANT.

[**Heredity of iron castings.**] L. GUILLET (Compt. rend., 1927, **185**, 1598).—More precise methods for the determination of the properties, and especially of the chemical compositions, of the castings described by Le Thomas (cf. preceding abstract) are required, in order to detect any small differences between them.

J. GRANT.

Hardness of chromium-nickel steels in relation to thermal treatment. P. J. SALDAU and V. N. SEMENOV (Ann. Inst. Anal. Physico-Chim. Leningrad, 1926, **3**, 181—210).—Comparison of the results of examination by the bullet-shield method of a steel containing 0.29% C, 5.08% Ni, and 1.79% Cr, and corresponding in structure with the martensite type of chromium-nickel steel according to Guillet's classification (B., 1906, 539 A), with those of Guillet (*loc. cit.*), Grenet, Rogers (B., 1919, 774 A), and Greaves (B., 1919, 821 A) shows that this steel corresponds also with Grenet's type III ("Trempe, recuit, cementations et conditions d'emploi des aciers," 1911), and is characterised by the fact that its transformation temperature during cooling (even very slow) differs greatly from that during heating. After heating to 650—680° and subsequent tempering in cold water, the steel exhibits minimum hardness and brittleness, and is easily worked in the cold. If heated to 650—680° and subsequently cooled slowly, it possesses a hardness near to that of the tempered metal and maximum brittleness, and is readily worked in the cold. The brittleness of the slowly cooled steel may be annulled by again heating to 650—670° and then quenching in cold water. On tempering during heating, the hardness remains almost constant as the temperature is raised to 680°, and afterwards increases rapidly to a maximum at 900°.

T. H. POPE.

Determination of manganese in steel by the Proctor-Smith reaction in presence of phosphoric acid. B. C. MUKERJEE (Analyst, 1927, **52**, 689—692).—A survey of previous methods for the determination of manganese in steel by the use of phosphoric acid is followed by a study of the influence on the process of temperature, velocity of titration, and concentration of acid. The solutions were standardised under the same conditions as in the actual analysis. Values for mangan-

ese in 7 steels were obtained and compared with those obtained when nitric acid alone was present in solution, and by methods other than the persulphate method. It was found that the use of nitric acid alone did not give higher results than if a mixture of nitric acid and sulphuric acid was used for dissolving the steel, and that the optimum condition for titrating manganese in the presence of phosphoric acid is for 1 c.c. of the phosphoric acid solution to contain 0.047 g. of phosphoric acid, for with higher concentrations slightly higher results are obtained, and with lower the end-point is not so sharp. D. G. HEWER.

Calculation of the combustion temperature in blast furnaces. K. MÜHLBRADT (Stahl u. Eisen, 1927, **47**, 1813—1816).—A theoretical discussion of the mechanism of the combustion of coke in a blast furnace, with special reference to the temperature distribution. In the blast furnace the reaction takes place between heterogeneous phases, hence all calculations of the initial temperature of the glowing carbon based on the principles of gas combustion lead to erroneous results. Thermodynamic considerations of the equilibria in the furnace indicate that there are two initial temperatures very different from one another, one due to the reaction in the solid phase and one to that in the gaseous phase, and that the temperature in any zone of the furnace is dependent on the extent to which each reaction takes place in that zone, modified by the effects of convection, conduction by the hot blast, and radiation. This theory is shown to account for observed facts in the distribution of temperature, effect of varying wind pressure, and reactivity of the coke. A. R. POWELL.

Differentiation of aluminium alloys based on the use of p_H indicators. C. QUILLARD (Compt. rend., 1927, **185**, 1281—1283).—The properties of aluminium alloys may be studied from a consideration of the colorimetric change in p_H value of twice-distilled water, or of a solution of pure sodium chloride (p_H 5.4), after immersion for 2 hrs. Alloys containing silicon, manganese, or magnesium have thus been classed in order of corrosion by the solution, and by the use of gelose to stabilise the colour-change at points of local attack it is shown that the "pure" American aluminium is attacked more than the ordinary French industrial product. Cleaning by means of ether and the sand-blast increased the attackability by water of both pure and industrial aluminums, but not always that of their alloys.

J. GRANT.

Volumetric determination of magnesium. J. VONDRÁK (Z. Zuckerind. Czechoslov., 1927, **52**, 113—119).—The method depends on the conversion of the magnesium salt into magnesium ammonium arsenate, reduction of this with sulphite, and titration with 0.1*N*-iodine at 37—50°; results 99—99.5% of the theoretical value were obtained. J. P. OGILVIE.

Spectroscopic determination of platinum in silver alloys. H. DE LASZLO (Ind. Eng. Chem., 1927, **19**, 1366—1368).—With a view of developing a rapid method of determining platinum in its ores the spectrographs of platinum-silver alloys were examined by photographing a condensed spark between a pair of the alloy electrodes with a quartz spectrograph. Standard

electrodes of alloys containing 5, 2, 1, and 0.5% Pt were made by bisecting platinum-silver beads prepared by cupellation and also by direct fusion. As the concentration of platinum decreases, certain of the spark lines of that metal vanish, and beads produced from an ore may be compared with the standard series. When prepared by the usual method of cupellation, beads showed lines associated with lead in every case.

C. A. KING.

Surface tension of metals with reference to soldering conditions. A. W. COFFMAN and S. W. PARR (Ind. Eng. Chem., 1927, 19, 1308—1311).—An adaptation of the apparatus used in the drop-weight method for determining the surface tension of liquids *in vacuo* was used. The average value for mercury at 25—27° was 468 dynes/cm., a variation of 1 to 1.5% from values obtained by previous investigators. The observed values for lead and tin at 350° were 446 and 524, respectively. Addition of lead to tin causes a pronounced fall in the surface tension of tin, whilst small quantities of tin added to lead reduce the value only slightly and then raise the surface tension until the value for pure tin is reached. A decided lowering of surface tension was noted when determined in an atmosphere of hydrogen chloride and a slight reverse effect in nitrogen. It is considered probable that a marked decrease in surface tension, and thereby a good spreading effect, would result from alloying two metals of widely differing atomic volumes and surface tensions, *e.g.*, alloys of bismuth and lead, antimony and lead, thallium with antimony, bismuth, or tin. The mechanism of the spreading of solder appears to follow the decrease in the surface tension or work of cohesion, and by spreading and alloying with the base metal, the solder further decreases the surface tension, and still further spreading takes place.

C. A. KING.

Action of fluxes in soft soldering and a new class of fluxes for soft soldering. R. S. DEAN and R. V. WILSON (Ind. Eng. Chem., 1927, 19, 1312—1314).—The spreading of soft solder on copper under the influence of various fluxes was determined by measuring the thickness of the globule of solder when melted at a definite temperature (200°) on a strip of copper. Gaseous hydrochloric and hydrobromic acids are among the best soldering fluxes, other good fluxes being compounds which evolve halogen or halogen-acid gases on heating. It is considered that a good flux may be found amongst the halogenated hydrocarbons of high mol. wt., *e.g.*, hexabromoethane. Naphthalene tetrachloride is in satisfactory use for certain types of commercial work.

C. A. KING.

Determination of iodine in minerals. E. WILKE-DÖRFURT (Z. angew. Chem., 1927, 40, 1478—1479).—The mineral is heated with sulphuric acid at 220—230° for 3 hrs. in a current of air or other suitable gas. The stream is passed through a receiver in which the iodine and hydrogen iodide are absorbed, and the iodine determined as usual. The process has advantages over the older alkaline-digestion method, *e.g.*, it requires only a third of the time, a smaller weight of mineral may be used, and the disturbing effect of silicic acid in the iodine titration is avoided; but with minerals containing much calcium carbonate the acid digestion gives rise to a

troublesome froth. The results given by the two methods are in close agreement. Certain superphosphate fertilisers, when examined by this method, were found to contain on the average 25 mg. of iodine per kg.

W. J. POWELL.

Chromium plating. F. R. PORTER (Brass World, 1927, 23, 267—269).—The bath contains chromic acid (120—240 g. per litre), basic chromic carbonate (7.5—15 g. per litre), chromic sulphate (3—7.5 g. per litre), and is used at 40—46°. Plane surfaces are plated at 70—350 amp. per sq. ft., and irregular surfaces at 200—250 amp. per sq. ft. Chromium plate is applied to iron over copper or nickel plate, since iron plated with chromium only is not protected from rusting.

CHEMICAL ABSTRACTS.

Spinning-centrifuge corrosion. EGGERT.—See V.

PATENTS.

Reduction of iron ores. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 280,976, 19.6.26. Addn. to B.P. 278,167; B., 1927, 942).—The process of the prior patent is modified in that, instead of using a reducing gas derived from powdered fuel, those prepared from gases or vapours containing hydrocarbons, *e.g.*, natural gas, coke-oven gas, or gases resulting from cracking processes, and oxygen or air rich in oxygen are used, the hydrocarbons being converted mainly into carbon monoxide and hydrogen.

M. E. NOTTAGE.

[Cast iron] alloy. H. WADE. From INTERNAT. NICKEL Co. (B.P. 281,051, 4.10.26).—Cast iron, highly resistant to oxidation and corrosion, contains 5—35% Ni and 2—15% Cr, nickel being present in the greater proportion. Other metals may be incorporated, *e.g.*, 0.3—10% Mn, up to 3% Al, and copper which in conjunction with nickel may be from 5% to 60%. Carbon (2—4%) is within the range of ordinary cast irons.

C. A. KING.

Purification of iron alloys with a high silicon content. G. F. JAUBERT (F.P. 619,561, 30.11.23).—Ferrosilicon alloys for use in the preparation of hydrogen for airships are ground wet and treated with small quantities of acid or alkali until a test of the residue shows that the sulphides, arsenides, and phosphides have been decomposed.

A. R. POWELL.

Acid or sea-water resisting [aluminium-tin] alloy. T. M. DIVEN (U.S.P. 1,652,301, 13.12.27. Appl., 25.3.27).—An alloy containing at least 93% Al, and approximately 5.25% Sn and 0.75% P.

F. G. CROSSE.

Solder for aluminium and its alloys. M. GORMANN (F.P. 619,236, 25.11.25).—Tin, zinc, aluminium, and silver are melted under a layer of lithium chloride and the resulting alloy is mixed with phosphorus, chromium, potassium chloride, and potassium pyrosulphate.

A. R. POWELL.

Production of magnesium from magnesia compounds. W. KOELLER (U.S.P. 1,650,893, 29.11.27. Appl., 27.1.26).—The magnesium compound is intensely heated at a reacting temperature in a current of hydrogen which is itself heated at the same temperature. The vapour of metallic magnesium thus produced is carried out of the furnace by the excess hydrogen and condensed in the gas.

M. E. NOTTAGE.

Production of magnesium from dolomite. W. KOEHLER (U.S.P. 1,650,894, 29.11.27. Appl., 8.2.26).—A charge containing magnesium and calcium compounds together with carbonaceous matter is heated in an electric furnace in a current of a reducing atmosphere until reacting conditions are obtained; calcium carbide and metallic magnesium are formed and the latter is removed from the furnace. M. E. NOTTAGE.

Operation of fume furnaces [for zinc]. G. H. WARING, Assr. to W. G. WARING (U.S.P. 1,652,224, 13.12.27. Appl., 4.6.25).—A fume furnace containing a replenished bed of zinciferous material, below which is a supply of primary air and above which is secondary air, is replenished by introducing a charge of zinc-bearing materials into the furnace at a point above the point of entry of secondary air while fine charge materials are tumbled in through the lateral walls of the furnace so as to scatter the charge through the ascending gases. M. E. NOTTAGE.

Extraction of zinc. J. SKOGMARK, Assr. to M. F. CHASE (U.S.P. 1,652,184, 13.12.27. Appl., 9.4.25).—In a blast furnace working zinc materials, part of the products of combustion are by-passed from a lower zone to the upper part of the furnace to preheat the descending charge. Gases carrying zinc vapours are withdrawn from an intermediate zone of the furnace. C. A. KING.

Manufacture of zinc. H. E. COLEY (B.P. 281,129, 2.2.27).—Preheated zinc ores in a comparatively thin layer are brought into contact with a reducing agent which is maintained cool and without decomposition up to the point of contact with the ore. A convenient method is to heat the ore in a rotary kiln, and maintain it at 930–1100° in a second rotary furnace into which crude oil, coal, etc. is introduced. C. A. KING.

Application of zinc coatings to iron articles. T. LIBAN (B.P. 257,262, 17.8.26. Ger., 18.8.25).—The articles are dipped, while still wet from the cleaning solution, into a bath of molten zinc (which may, if desired, contain aluminium) on which floats a protective layer of fused salts consisting of fluorine compounds (*e.g.*, alkali fluoride or fluorobenzene) or double salts of fluorine mixed with zinc chloride, with or without ammonium chloride. By their passage through this layer, the articles are dried, preheated, and further cleaned. M. E. NOTTAGE.

White-gold solder. T. P. SHIELDS, Assr. to SHIELDS & MOORE (U.S.P. 1,652,740, 13.12.27. Appl., 24.7.25).—The alloy chiefly contains 40–82% Au, together with nickel, zinc, and silver in the proportions of 4–10 pts. of nickel, 8–20 pts. of zinc, and 10–25 pts. of silver. F. G. CROSSE.

Coating a body with platinum. S. G. S. DICKER. From N. V. PHILIPS' GLOBILAMPENFABR. (B.P. 280,697, 29.10.26).—The object to be coated is heated in an atmosphere of a volatile platinum compound, *e.g.*, "carbonyl platinous chloride." J. S. G. THOMAS.

Preparation of metals with high-temperature fusing points, such as tungsten, and of wire therefrom. F. KOREF and H. ALTERTHUM, Assrs. to GEN. ELECTRIC CO. (U.S.P. 1,650,631, 29.11.27. Appl., 6.1.23. Ger., 17.8.22).—Powdered tungsten is compressed,

sintered into a large crystal in an atmosphere of reducing gas, and worked while still hot into a flexible and ductile state. M. E. NOTTAGE.

Separation of rocks and minerals from clay gangue. ANC. ÉTABL. BARBIER, BÉNARD, ET TURENNE (Soc. ANON.) (F.P. 621,829, 21.9.26).—The mass is heated in an atmosphere of steam at 150° under pressure, and the clay gangue then removed by a washing process. A. R. POWELL.

Production of metals from oxide ores. E. G. T. GUSTAFSSON (B.P. 269,179, 6.4.27. Swed., 10.4.26).—Metals such as iron and iron alloys, zinc, copper, etc. may be obtained by reduction of the oxide ore with fine-grained charred or coked carbonaceous material; this is obtained from waste wood or bituminous coals either by treatment in a separate charcoal or coke oven or by making use of the gases from the reduction chamber. M. E. NOTTAGE.

Chlorinating roasting of burnt ore. H. STEPHAN (B.P. 262,392, 1.10.26. Ger., 4.12.25).—In the chlorination of burnt ores, materials are added to the charge which themselves evolve water or by interaction produce water at temperatures approximating to that of roasting, *e.g.*, ferric hydroxide, crystallised sodium or magnesium sulphate, and hydrochloric acid, the last of which, by interaction with sulphides and oxidation of the hydrogen sulphide formed, produces water vapour. C. A. KING.

Testing the hardness of metals. J. C. B. FIRTH and F. G. DICKINSON (B.P. 280,292, 12.8.26).—Impressions are made on a metal by a ball or diamond through the medium of a loaded spring located within a cylinder which is capable of vertical movement inside an outer cylinder also movable. The load on the spring is determined at a given point in the relative movement of the inner and outer cylinders, the further forward movement of the cylinders being arrested. The setting of the load may be varied or locked. C. A. KING.

Bright annealing furnace. SIEMENS-SCHUCKERTWERKE A.-G., Asses. of SIEMENS-SCHUCKERTWERKE G.M.B.H. (B.P. 276,674, 25.8.27. Ger., 26.8.26).—After their passage through a pipe contained in a water-bath, bands and wires are freed from adhering water by passing them into a stove where they are heated to a temperature a little above 100°, then through a condenser, where the water is removed, and thence into the furnace. M. E. NOTTAGE.

Mineral separator. D. STEWART & CO., LTD. From W. MAUSS (B.P. 280,739, 18.1.27).—A cylindrical perforated drum rotates on a vertical hollow shaft inside a surrounding container. Pulp is fed down the shaft to the drum and the heavier constituents pass by centrifugal action from the drum into a non-rotating liquid in the outer container, the separation being effected partially by centrifugal force and partially by gravity. C. A. KING.

Bi-metallic thermostat material. P. H. BRACE, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,652,556, 13.12.27. Appl., 12.3.25).—The two elements of the thermostat consist of nickel-steel alloys, the nickel/iron ratio in which is less than 0.3 and more than 0.3, respectively. F. G. CROSSE.

Chemical process for welding of metals. B. RACHIEFF and M. GOFMANN (B.P. 274,481, 14.7.27. Fr., 15.7.26).—An intimate mixture of calcium, aluminium, and the metal to be welded is placed between the parts of a metal and the whole raised to about 700° to initiate combustion of the mixture, which attains such a temperature as to fuse and weld the parts. About 2 pts. of calcium are mixed with 3 pts. of aluminium, and 1–2 pts. of this mixture are incorporated with 8–9 pts. of the metal to be welded. C. A. KING.

Production of metallised surfaces on non-metallic bodies. A. I. G. WARREN, and PRECIOUS METAL INDUSTRIES, LTD. (B.P. 280,651, 24.8.26).—Non-metallic materials, *e.g.*, bakelite, casein and bituminous products, etc., are impregnated with sulphur or a sulphur compound and treated with a metallic salt so as to form a coating of a metallic sulphide which is then reduced to metallic form. Further metal may be deposited electrolytically on the film of metal on the non-conducting article. C. A. KING.

Nickel-plating bath. COMP. IND. DE MÉCANIQUE HORLOGÈRE (F.P. 619,615, 8.12.25).—A nickel-plating bath contains a soluble nickel salt, an alkali fluoride or fluosilicate, and a weak acid, *e.g.*, boric acid. A quantity of a cobalt salt is preferably added or a cobalt anode may be used. The deposits obtained are equal to those produced in a fluoroborate bath, but the above bath is more stable. A. R. POWELL.

Electrodeposition of chromium. L. MELLERSH-JACKSON. From SIEMENS & HALSKE A.-G. (B.P. 266,379, 19.2.27).—The temperature of the electrolytic bath is controlled to within 1° by means of a suitable medium, *e.g.*, water or steam, flowing through a hollow anode of lead or lead-containing material resistant to the action of the electrolyte. J. S. G. THOMAS.

[Anode for] electrolytic deposition of chromium. GEN. MOTORS CORP., Assecs. of W. M. PHILLIPS (B.P. 273,659, 18.3.27. U.S., 30.6.26).—A ferrous metal anode, more especially one of low-carbon steel, constituting a container for the bath, is employed in the electrolytic deposition of chromium from a bath containing chromic acid and preferably not more than 12½ g./litre of chromium sulphate. J. S. G. THOMAS.

Chromium plating. METALS PROTECTION CORP., Assecs. of C. H. HUMPHRIES (B.P. 277,295, 24.6.27. U.S., 9.9.26).—A dull chromium-plated article plated at about 20° is given a bright or satin finish by further plating at about 40°. J. S. G. THOMAS.

Electrolytic separation of metallic chromium for the preparation of chromium coatings on other metals. R. APPEL (B.P. 274,882, 20.7.27. Ger., 21.7.26).—Chromium is electrolytically deposited from a bath of chromic acid solution containing not more than 0.5% of one or more borates or iodates of the alkali metals per litre. J. S. G. THOMAS.

Electrolytic preparation of magnesium and metals of the alkaline earths. A. C. JESSUP (B.P. 259,554, 27.9.26. Fr., 9.10.25).—The following layers are superposed in a gravity electrolytic cell: (1) a layer of impure metal or alloy, *e.g.*, lead-magnesium alloy,

serving as anode; (2) above this a mixture of suitable density of chloride of the metal to be obtained with one or more metallic chlorides of a more electropositive metal, *e.g.*, barium chloride; (3) a layer of pure metal, *e.g.*, magnesium, serving as cathode, and (4) at the top a protective covering of light chlorides, *e.g.*, a mixture of potassium chloride and magnesium chloride in the proportions $5\text{KCl} + \text{MgCl}_2$. J. S. G. THOMAS.

Production of aluminium in electric furnaces. SOC. ITAL. DI ELETTROCHIMICA (B.P. 274,108, 8.7.27. Italy, 8.7.26).—The agglomeration of aluminium hydroxide, either by the addition of suitable substances such as gelatin, glue, colloids, acids, salts, etc., preferably in solution and/or by compressing it at a suitable temperature, is claimed. The product is then dried, preferably in an electric furnace. M. E. NOTTAGE.

Manufacture of electromagnetic cores. I. G. FARBENIND. A.-G. (F.P. 619,290, 26.7.26. Ger., 19.8.25 and 5.7.26).—Powdered iron, cobalt, or nickel, or a mixture of these produced by thermal decomposition of the carbonyls, is mixed with a binding material, *e.g.*, alcoholic lacquer solutions, and the mixture is pressed into shape under a high pressure. The products have a high permeability. A. R. POWELL.

Magnetic materials [alloys]. WESTERN ELECTRIC CO., INC., Assecs. of G. W. ELMEN (B.P. 273,638 and 274,136, [A] 16.8.26 and [B] 21.8.26. U.S., [A] 30.6.26 and [B] 12.7.26).—(A) A magnetic alloy having a negligible variation (less than 0.1% from 0 to 100 c.g.s. units) in permeability over a wide range of flux densities contains approximately 45% Ni, 25% Co, and 30% Fe. (B) The introduction of molybdenum into a magnetic element, *e.g.*, nickel, increases the initial permeability and is consistent with a low hysteresis loss and high resistivity. Such an alloy contains approximately 94% Ni and 5% Mo. C. A. KING.

XL—ELECTROTECHNICS.

Electrical heating equipment for chemical work. G. ANGEL (Ind. Eng. Chem., 1927, 19, 1314–1316).—For heating or evaporating corrosive liquids in vessels, *e.g.*, glass, quartz, stoneware, which are of necessity non-conductors, two electrodes of graphite or other suitable material, surrounded by pipes of stoneware or fused quartz glass, are used. After the current is turned on, the pipes quickly become filled with vapour so that the electrodes are in contact with the liquids only at their ends, and owing to the high current density lighting arcs begin to burn. Electrodes of graphite were used for heating concentrated hydrochloric acid in quartz vessels, and for sodium sulphate and sodium chloride solutions iron was sufficiently resistant both for electrodes and tanks. For the external heating of caustic pots a bank of electrically-heated nichrome coils was hung vertically around the pot, the whole being carefully insulated to prevent loss of heat by radiation. The energy efficiency of this furnace was about 86%. C. A. KING.

Ozone formation. SAUNDERS and SILVERMAN.—See VII.

Chromium plating. PORTER.—See X.

PATENTS.

Electric resistance furnace. A. J. HANSON, Assr. to AMER. METALLURGICAL CORP. (U.S.P. 1,652,200, 13.12.27. Appl., 23.1.21).—An insulated ribbon resistor is supported upon the furnace-chamber wall so that there is an air space around a substantial portion of the resistor, and the insulation and support are provided with an imperforate covering. J. S. G. THOMAS.

Electric furnace. J. M. WEED, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,650,675, 29.11.27. Appl., 23.3.26).—A crucible, forming a reservoir for the charge and divided into a number of separate parts connected together at their bottoms through a duct having contiguous legs in parallel planes, is provided with means for passing an electric current through a portion of the charge in the duct. J. S. G. THOMAS.

[Metal-melting] electric furnace. J. A. SEEDE, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,650,658, 29.11.27. Appl., 5.6.26).—A metal-melting furnace having a number of electrodes above the charge is provided with transformer means comprising primary coils connected with a three-phase source of current, and secondary coils joined in star formation and connected with the upper electrodes. The neutral point of these secondary coils is connected with the bottom of the furnace, and means are provided for reversing the connexions of one of the primary coils so that the polarity of one of the secondary coils is reversed. J. S. G. THOMAS.

High-frequency electric induction [vacuum] furnace. N. R. DAVIS and METROPOLITAN-VICKERS ELECTRICAL Co., LTD. (B.P. 281,379, 31.8.26).—The radius and height of the tubular chimney or cover of a high-frequency electric induction vacuum furnace are so related to the frequency of the current supply that serious glow discharge in the metallic vapour heated in the furnace is prevented. J. S. G. THOMAS.

[Protecting resistors in] electrical resistance furnaces. METALLWERK PLANSEE G.M.B.H., and P. SCHWARZKOPF (B.P. 272,933, 15.6.27. Ger., 16.6.26).—Oxidation of resistors of molybdenum or tungsten or their alloys is prevented by embedding the resistors in a layer of wood charcoal or other non-conducting charcoal, to which may be added non-conducting oxides of high m.p. Charcoal consumed during operation of the furnace is replaced through a feed-hopper. J. S. G. THOMAS.

Control for electric furnaces. E. C. GLITZKE (U.S.P. 1,650,624, 29.11.27. Appl., 19.11.26).—In an electric furnace a control for limiting its normal working temperature consists of a heating element which is connected in an electric circuit. A closure is also provided which is fitted with terminals connected in the electric circuit by means of a fusible element having a predetermined m.p., and which extends into the furnace to receive the same heat as the material undergoing treatment. M. E. NOTTAGE.

Electrolytic cell. A. K. CROAD, From JESSUP & MOORE PAPER Co. (B.P. 281,184, 2.6.26).—An electrolytic cell comprises a container, perforated basket electrode, gas chamber, take-off pipe from gas chamber, and a vent from the space between electrode basket and container, adapted to provide a maximum cell capacity per unit cell volume. J. S. G. THOMAS.

Production of electric lead accumulators. F. ALETTER (B.P. 268,366, 24.3.27. Ger., 29.3.26).—The charged electrodes of lead accumulators are discharged in acid until their effective mass is converted completely or for the greater part into lead sulphate, and are then recharged. Batteries having plates so treated are filled with distilled water containing, if desired, a little conductive salt, e.g., sodium sulphate, or may contain a dry filling in which there is little or no acid, to which distilled water is added before use. J. S. G. THOMAS.

Manufacture of anodes. A. P. MUNNING & Co. (F.P. 617,451, 11.6.26).—Circular plates, perforated in the centre, are made from sheets of electrolytically-prepared metal, e.g., nickel, and mounted on a rod. The whole anode surface can then be uniformly exposed to the electric current. J. S. G. THOMAS.

Electrodes for electrolytic cells. R. PECHKRANZ (B.P. 265,606, 4.2.27. Switz., 5.2.26).—A thin plate of metal, other than a precious metal, is rigidly fixed to a frame which is thicker than the plate and is provided with recesses forming conduits for water and gases. J. S. G. THOMAS.

Production of a lead product. [Recovery of lead from storage battery waste.] J. O. HANDY, Assr. to PRIMOS Co. (U.S.P. 1,651,886, 6.12.27. Appl., 26.9.24).—Brittle parts of storage battery waste are mechanically dislodged and separated from the non-brittle parts, and treated in the cold with an alkali carbonate solution. The residue separated from the resulting mixture is roasted. J. S. G. THOMAS.

Manufacture of fireproof electric insulated cables. C. B. BACKER (U.S.P. 1,650,972, 29.11.27. Appl., 28.9.26. Norw., 9.10.25).—An electric conductor of relatively large diameter is enclosed within a tubular member of much larger diameter, the space between being loosely filled with metallic magnesium. The whole is subjected to steam at relatively high pressure until the magnesium has been completely converted into magnesium oxide. The cable is then subjected to drawing operations as required. M. E. NOTTAGE.

Evacuation of bulbs and other vessels. D. S. GUSTIN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,651,386, 6.12.27. Appl., 26.5.22).—The bulb is flushed with oxygen and connected with a source of vacuum containing phosphorus. J. S. G. THOMAS.

Electrode holders for electric furnaces. NORSKE A./S. F. ELEKTROKEM. IND., Assees. of A.-G. F. STICKSTOFFDÜNGER (B.P. 262,481, 7.12.26. Ger., 7.12.25).

Photo-electric cell. TELEFUNKEN GES. F. DRAHTLOSE TELEGRAPHIE M.B.H., and A. KAROLUS (B.P. 264,173, 7.1.27. Ger., 8.1.26).

[Plates for] galvanic batteries. A. LEWIS (B.P. 281,018, 26.8.26).

Platinum coatings (B.P. 280,697). **Chromium plating** (B.P. 266,379, 273,659, 277,295, and 274,882). **Preparation of magnesium and alkaline-earth metals** (B.P. 259,554). **Electromagnetic cores** (F.P. 619,290). **Magnetic materials** (B.P. 273,638 and 274,136).—See X. **Purification of sugar solution** (B.P. 272,943).—See XVII. **Sterilising liquids** (B.P. 257,956).—See XIX. **Photographic plates or films** (B.P. 275,933).—See XXI.

XII.—FATS; OILS; WAXES.

Determination of the acid and saponification value of dark-coloured fats. H. DUBOVITZ (Chem.-Ztg., 1927, 51, 984).—To determine the saponif. value 1 g. of the fat is heated with 50 c.c. of a 0.2% alcoholic solution of potassium hydroxide for 1 hr. on the water-bath whereby the fat is partially saponified. After cooling and adding 10 c.c. of 10% potassium chloride solution and 40 c.c. of water, the unsaponifiable matter and the unsaponified fat are extracted with light petroleum, from which they are recovered by evaporation and weighed. The loss in weight is the amount of fat saponified by the potassium hydroxide used. The acid value is determined by dissolving 0.3—1 g. of fat in hot neutral alcohol, cooling, and adding standard alkali until the solution darkens with thymolphthalein, extracting the remaining fatty acid with light petroleum, and weighing it after distilling off the solvent; the loss in weight gives the amount of free fatty acid. The method does not give good results if the fat contains unsaponifiable matter insoluble in light petroleum.

A. R. POWELL.

General interpretation of the drying processes of fatty oils. B. SCHEIFELE (Farben-Ztg., 1927, 33, 739—741).—The drying processes of all drying oils, whether of the tung oil, linseed oil, or poppyseed oil groups, are explained with reference to the relative unsaturation of their molecules and consequent tendency to gel formation.

S. S. WOOLF.

Accidental green coloration of olive oils. R. MARCILLE (Ann. Falsif., 1927, 20, 538—539).—The green colour assumed by olive oil on keeping in vessels of tinned iron or on boiling with distilled water is shown to be due to the presence of copper, derived from the metal or the distilled water, which yields a strongly coloured compound with the chlorophyll. The reaction may be used for the detection of traces of copper in water by boiling 200—250 c.c. of the water with about 10 c.c. of olive oil for 10 min. and examining the colour of the oil.

F. R. ENNOS.

Deodorising menhaden fish oil. E. E. RANDOLPH (J. Elisha Mitchell Sci. Soc., 1926, 42, 131—134).—The odour, which is ascribed to the presence of an acid, $C_{18}H_{38}O_2$, is removed by heating the oil with formaldehyde and hydrochloric acid, followed by treatment with superheated steam.

CHEMICAL ABSTRACTS.

Explosions with ether. BRANDT.—See XX.

PATENTS.

Extraction of oil from the pericarp of palm fruit and like nuts. C. DOWNS and R. A. BELLWOOD (B.P. 280,986, 21.7.26).—The pericarp of palm fruit or similar nuts is stripped by mechanical agitators of irregular surface and subjected to the action of hot solvent in suitable apparatus. Preliminary steaming of the fruit is dispensed with.

S. S. WOOLF.

Emulsification of vegetable and animal oils, fats, greases, waxes, etc. W. E. BILLINGHAME (B.P. 280,762, 25.5.26).—A concentrated emulsion, prepared by blending a small amount of vegetable or animal oil, fat, grease, wax, etc. with saponaceous ingredients, e.g., rosin soaps, and soluble proteins, is

used as emulsifying agent for the bulk of the oil to be treated, the mixture being subsequently diluted with water. [Stat. ref. to B.P. 13,151 of 1905, 11,028 of 1903, 8141 of 1900, and 252,449.] S. S. WOOLF.

Production of emulsions. W. A. WHATMOUGH (B.P. 280,096, 13.5.26).—A fatty oil to which is added a small quantity of a higher and non-volatile fatty acid and, if desired, a hydrocarbon product, e.g., paraffin jelly, is mixed with water containing a protein and sufficient alkali to reduce the interfacial tension between the oil and water phases sufficiently to permit rapid emulsification in the cold. To emulsions so produced neutral solutions of an essentially mineral salt, fruit or vegetable juice, and/or a syrup or sugar solution, additional fatty acid, or waxes may be added. The whole may be evaporated at a low temperature under vacuum in order to remove air bubbles and excess of water and to stabilise the emulsion.

S. S. WOOLF.

Manufacture of toilet creams. J. C. & J. FIELD, LTD., and E. POLAN (B.P. 281,425, 24.9.26).—Toilet creams, particularly shaving creams, are prepared from a fatty acid, a fatty oil or fat (for part of which a mineral oil may be substituted), glycerin, and ammonia solution. The ammonia is not more than that equivalent to the fatty acid, and the cream is alkaline to phenolphthalein when stirred with water. Part of the ammonia may be substituted by sodium or potassium hydroxide. Directions are given for the production of a typical cream from stearic acid, olive oil, glycerin, and ammonia solution.

B. FULMAN.

Apparatus for heating oleaginous materials. F. KRUPP GRUSONWERK A.-G. (B.P. 259,939, 25.9.26. Ger., 17.10.25).

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Significance of negative catalysis in the formation of protective coatings. J. SCHEIBER (Farben-Ztg., 1927, 33, 680—682).—The drying of tung oil differs from that of linseed and poppy-seed oils very markedly, owing to the high gelatinising tendency of the first, autoxidation playing a more pronounced part in the drying of the others. It follows that the action of driers as oxidation catalysts will be correspondingly more significant in linseed and similar oils, this being supported by the fact that the drying of tung oil is affected by many substances, e.g., iodine, aluminium chloride, etc., that have no parallel effect on linseed oil. The oxidation function of driers on the linseed oil type, however, does not cease with film formation, but continues to the eventual detriment of the film. Of various methods available for combating this tendency, the introduction of slow-acting negative catalysts or "anti-oxygens," e.g., phenols, amines, hydroxylamine derivatives, carbamide, etc., has not received the attention warranted. The application of these negative catalysts is discussed and their use in mixed tung oil-linseed oil vehicles is recommended.

S. S. WOOLF.

The red lead question. H. RAHDER (Farben-Ztg., 1927, 33, 741—743).—The advantages claimed for modern "high-dispersion" red leads are: non-settling, easy brushing, good spreading power and consequent cheapness. The author complains, however, that no

attention has hitherto been paid to questions of actual rust prevention, and his preliminary tests indicate that in adhesion, resistance to water, etc. the "modern" red lead is inferior to the "classic." S. S. WOOLF.

Hygienic properties of titanium dioxide and "titanium white." K. B. LEHMANN and L. HERGET (Chem.-Ztg., 1927, 51, 793—794).—Titanium dioxide proved harmless when administered to cats, rabbits, etc. over periods up to 16 months, and titanium white is considered to be a perfectly non-poisonous pigment. Methods for the analytical determination of titanium are described. S. S. WOOLF.

Hardening and esterifications of rosin. E. PYHÄLÄ (Farben-Ztg., 1927, 33, 801—803).—The author describes a new process for the esterification of rosin in open kettles, the customary losses and darkening being avoided. Finely-divided calcium compounds of the appropriate higher alcohols, phenols, etc. are gradually added, with stirring, to molten rosin. The temperature is maintained at 180—220° until effervescence ceases and a calm melt is obtained. By careful working a pure ester is produced, lime being deposited quantitatively as a sediment in the kettle. Experimental details are given for the preparation of esters of rosin with glycerol (mono- and tri-glycerides), phenol, cresol, β -naphthol, resorcinol, and borneol. S. S. WOOLF.

Resins in petroleum. SACHANEN and WASSILIEV.—See II. **Drying of fatty oils.** SCHEIFELE.—See XII.

PATENTS.

Manufacture of colloiddally dispersed white lead. T. GOLDSCHMIDT A.-G. (G.P. 444,431, 3.10.22).—Carbon dioxide and water are allowed to react with lead oxide which is in the desired state of division, or which is prepared from solid lead compounds *in situ* by thermal decomposition (e.g., of lead carbonate, oxalate, or nitrate) or by interaction with fluid media.

C. HOLLINS.

Manufacture of red lead. E. BARTHELMESS (G.P. 443,853, 20.3.23).—Lead monoxide or carbonate is fed continuously into a revolving furnace packed so as to minimise heat losses.

C. HOLLINS.

Manufacture of light-fast lithopone or zinc sulphide. E. MAASS and R. KEMPF (G.P. 444,432—3, [A] 10.5.23, [B] 26.8.24).—(A) The crude lithopone is treated, cold or hot, with compounds containing loosely-bound sulphur or easily dissociating with production of sulphide ions (e.g., metal sulphides, hydrosulphides, or polysulphides), and is then washed until neutral to phenolphthalein, dried, calcined, and chilled in the usual way. (B) The lithopone before calcination is treated first with a small proportion of a soluble thiosulphate, polythionate, sulphite, bisulphite, hyposulphite, etc. and an ammonium salt, then with a sulphur-free acid-binding agent, e.g., baryta, barium carbonate, sodium carbonate, and finally washed.

C. HOLLINS.

White [titanium] pigment. B. LAPORTE, LTD., H. E. ALCOCK and I. E. WEBER (B.P. 280,435, 27.4.27).—3 pts. of titanium hydroxide are mixed with 7 pts. of *blanc fixe* (made as described in B.P. 252,768; B., 1926, 666), both being in the form of pastes in water, carbon or some carbonaceous material, e.g., starch, sugar, oil,

etc., is added, and the mixed mass is heated at 700—950° until the carbon has completely burnt off. After cooling, the residue is ground for use as a white pigment.

S. S. WOOLF.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Physico-mechanical properties of vulcanised rubber at high temperatures. II. A. VAN ROSSEM and H. VAN DER MEYDEN (Kautschuk, 1927, 364—371; cf. B., 1926, 502).—Rubber vulcanised to a coefficient of approximately 3 does not always exhibit almost immediate low tensile strength at 147°: the development of brittleness at 147° with various samples of rubber occurs at widely different coefficients of vulcanisation, rubbers with a longer "optimum cure" becoming brittle more slowly and at a higher vulcanisation coefficient. For samples of low degree of vulcanisation, rise of temperature causes a decrease in the hardness as indicated by a "durometer," whereas at higher degrees of vulcanisation it causes a small increase. At 147° vulcanised rubber becomes plastic to the influence of pressure, but at 137° or 100° plasticity is observable only at very low degrees of vulcanisation. Lightly vulcanised rubber in a state of extension can be "frozen" by cooling to a sufficiently low temperature.

D. F. TWISS.

Colloid chemistry of rubber latices. III. Individual shape of the rubber particles and their heritability. E. A. HAUSER (Kautschuk, 1927, 357—359).—Amongst the globules in latex from individual trees there occurs a proportion of particles of characteristic shape. The characteristic type of globule can be observed in latex of any tree of one clone and also in the latex of the mother tree. In cases where the latex from a tree produced by bud-grafting fails to reveal the characteristic globules, their absence is attributed to the tree in question having inherited the features of the stock. Latex from a tree grown from the seed of a definite mother tree fertilised by pollen from a definite male tree generally shows two characteristic types of globule (of which one may predominate) corresponding with the individual particle shape for the two parent trees. In some latices the variation in the size of globule is small, e.g., 0.5—1.5 μ , whereas in others it may range from 0.5 to 4.0 μ ; the former type is generally derived from trees with smooth bark and the latter from trees with bark surface irregular and often showing signs of disease.

D. F. TWISS.

South African rubber. E. NEUFELD (Kautschuk, 1927, 359—364).—The production and properties of latex and rubber from *Euphorbia* trees are reviewed. *Euphorbia* latices contain vesicant substances. With the exception of *Euphorbia tirucalli* (the rubber of which is relatively inferior) and *Euphorbia dregeana*, the latices very easily undergo spontaneous coagulation. If a coagulant is required, a mixture of hydrochloric acid and tannin gives good results. For the preservation of *Euphorbia* latex, ammonia and formaldehyde are both effective. Vulcanised products containing a mixture of ordinary plantation rubber with partially deresinised *Euphorbia* rubber exhibit satisfactory mechanical properties, and the softness of the latter rubber facilitates the milling operation. The resins from *Euphorbia*

rubber are of promise for the manufacture of varnishes and insulating materials. D. F. TWISS.

PATENTS.

Manufacture of vulcanised rubber and materials for use therein. BRIT. DYESTUFFS CORP., LTD., C. J. T. CRONSHAW, and W. J. S. NAUNTON (B.P. 280,661, 3.9.26).—Although the condensation product obtained from acetaldehyde and α - or β -naphthylamine in the presence of an acid has no substantial influence on the rate of vulcanisation of rubber or on the ageing properties of the product, the condensation product of acetaldehyde with a mixture of these naphthylamines in equal proportions has a remarkable improving effect on the ageing properties. D. F. TWISS.

Rubber vulcanisation accelerator. C. O. NORTH, ASSR. to RUBBER SERVICE LABORATORIES CO. (U.S.P. 1,651,931, 6.12.27. Appl., 17.2.25).—Vulcanisation of rubber with sulphur is effected in the presence of a mixture of accelerators comprising the aldehyde derivative of dehydrated ethyldeneaniline and the aldehyde reaction product of this aldehyde derivative. D. F. TWISS.

Rubber compositions. RUBBER LATEX RES. CORP., ASSEES. of W. B. WESCOTT (B.P. 272,187, 14.5.27. U.S., 1.6.26).—Reinforced rubber, *e.g.*, for shoe soles, is made by treating fibre or rubber-containing fibrous scrap with rubber latex, drying the product, and milling it into a rubber composition containing not less than 50% of rubber. This method of procedure minimises damage to the fibre during the milling operation. D. F. TWISS.

Compounding of rubber. H. A. ENDRES, ASSR. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,652,141, 6.12.27. Appl., 15.9.23).—A solution of a coagulant containing a filler material which is peptised thereby is added to a suspension or emulsion of rubber; *e.g.*, rubber latex is coagulated by introduction into a mixture of sodium sulphate with excess of barium chloride. D. F. TWISS.

Coagulation of rubber latex. H. E. POTTS. From R. KOEPP & Co. (B.P. 280,639, 18.8.26).—Rubber latex is coagulated by means of sodium biformate alone or in conjunction with other coagulants. D. F. TWISS.

XV.—LEATHER; GLUE.

PATENTS.

Manufacture of white artificial horn. A. JÄGER, and HEROLD A.-G. (B.P. 281,537, 17.5.27).—In modification of B.P. 8875 of 1905 (B., 1906, 195), 1 pt. of phenol is condensed with 2.2–3 pts. of 30% formaldehyde solution, in the presence of 0.35 pt. of alkali salts of the fatty acids, preferably sodium stearate (or sodium hydroxide and stearic acid), at 71° for 90 min. After dehydration *in vacuo*, there are added 1.8 mols. of salicylic acid per mol. of alkali. After further dehydration, the mass is poured into open moulds to harden at 80–88°, yielding a white product, fast to air and light, the toughness of which is increased by adding small quantities of oily substances to the mix. B. FULLMAN.

Testing [the strength of] gelatinous substances. T. HALL and R. L. HOUTZ (U.S.P. 1,651,596, 6.12.27. Appl., 15.5.23).—A member of suitable weight and area

is supported in contact with the surface of the substance. The support is then removed to allow the member to depress the surface without penetrating it, and the movement of the member is measured. H. HOLMES.

XVI.—AGRICULTURE.

Effect of absorption by plants on concentration of soil solution. C. S. SCOFIELD (J. Agric. Res., 1927, 35, 745–756).—Experiments in water culture with barley and wheat seedlings show that these plants do not absorb water and dissolved substances in the proportions in which they are present in the solution. In 24 days the plants absorbed a quantity of water approximately equal to the original volume of the culture solutions, but at the end of this time the solutions contained 78–91% of the salts originally present. The bearing of the results on problems of irrigation is discussed.

C. T. GIMMINGHAM.

Determination of nitrogen in soils. C. OLSEN (Compt. rend. Trav. Lab. Carlsberg, 1927, 17, [3], 1–14).—A method is described suitable for the determination of the total nitrogen in soils where it is necessary to take into account the nitrogen present as nitrate and nitrite. 5 g. of the moist soil are placed in a Kjeldahl flask, and 10 c.c. of water (or of 5% potassium permanganate solution, if nitrites are present) and 5 g. of reduced iron are added, together with a few drops of octyl alcohol to prevent frothing. Then 30 c.c. of cold sulphuric acid (1 pt. of acid to 2 pts. of water by vol.) are added, and when vigorous effervescence has subsided, the mixture is heated gently for 45 min. The analysis is completed as in an ordinary determination of nitrogen by the Kjeldahl method. Full details are given. C. T. GIMMINGHAM.

Determination of the salinity of soils from the electrical conductivity of their aqueous extracts. N. K. VOSKRESENSKI (Ann. Inst. Anal. Physico-Chim. Leningrad, 1926, 3, 302–304).—The saline contents of 18 soils of various types are calculated from the conductivities of their clear aqueous extracts by means of the formula $M = C\kappa_{18} \times 10^6x$, where M represents the dry residue of the extract in mg. per litre, κ_{18} the specific conductivity of the extract diluted x times with distilled water to the concentration 0.002–0.003N, and C is a coefficient. In four cases, the extracts were characterised by low conductivities and by comparatively high content of organic matter, the latter amounting to 15–28% of the dry residue. For the remaining 14 extracts, the values of C differ from the mean value by 6% on the average and by 11% at most. There seems to be no simple relationship between the value of C and the content of humus in the soil.

T. H. POPE.

Relation of soil nitrogen to nodule development and fixation of nitrogen by certain legumes. G. GRÖBEL (New Jersey Agric. Exp. Sta. Bull., 1926, No. 436, 3–125).—With lucerne, infection and the establishment of nitrogen fixation proceed best in plants well supplied with combined nitrogen during the early stages of growth. Very little nitrogen was fixed during the first crop before the flowering period; the amounts of nitrogen fixed thereafter were inversely proportional to the amounts of soluble soil nitrogen at the disposal

of the plants. On soils depleted of nitrogen, gains in soil nitrogen resulted from the growth of six crops of inoculated lucerne when the tops were removed, but from soils well provided with available nitrogen, losses occurred. Somewhat similar results were obtained with soya beans. In both cases nodule development was adversely affected by the presence of large amounts of nitrate in soil or sand cultures. The effect appears to be connected with the nutrition of the plants, the nitrate being used in the first place to meet the need of nitrogen by the plant.

CHEMICAL ABSTRACTS.

Effect of potash fertiliser on the carrying quality of tomatoes. W. B. LANHAM (Texas Agric. Exp. Sta. Bull., 1927, No. 357, 38 pp.).—The presence of potash in the fertiliser increased the yield, but had no consistent effect on resistance to rough handling.

CHEMICAL ABSTRACTS.

Wheat. Influence of heredity and environment. F. T. SHUTT (Dep. Agric. Can., Rep. Dom. Chemist (1926), 1927, 62—70).—Data are recorded for the weight per 1000 kernels, and the water, nitrogen, and ash content, for 165 samples representing 45 varieties of wheat grown in 18 locations in Canada during 1924. The effect of the previous crop is recorded, together with data for the protein content of barley following various crops.

CHEMICAL ABSTRACTS.

Adaptation of certain colorimetric methods to the determination of nitrates, phosphates, and potassium in plant solutions. B. E. GILBERT (Plant Physiol., 1926, 1, 191—199).—The phenoldisulphonic acid method for nitrate, the ceruleo-molybdate method for phosphate, and the reduced chloroplatinate method for potassium were adapted to the analysis of tissue juices from maize, turnips, celery, beet, spinach, and lettuce. The results indicate the optimum nutrient levels of the three principal fertiliser elements.

CHEMICAL ABSTRACTS.

Sodium chlorate and other chemicals as herbicides for field bindweed. W. L. LATSHAW and J. W. ZAHNLEY (J. Agric. Res., 1927, 35, 757—767).—Field experiments with various chemicals (sodium chlorate, arsenite, and hypochlorite, zinc chloride, and copper sulphate) indicate that sodium chlorate is a satisfactory weed-killer for field bindweed (*Convolvulus arvensis*). A 12.5% solution applied when the plants are in full bloom was very effective, and had no permanent harmful effect on the soil. None of the other substances proved useful. Observations on the plant tissues suggested that sodium chlorate interferes with photosynthesis and compels the plants to exhaust the food reserves in the roots.

C. T. GIMMINGHAM.

Development of more effective dust fungicides by adding oxidising agents to sulphur. H. A. LEE and J. P. MARTIN (Science, 1927, 66, 178).—In the control of eye-spot disease of sugar cane by sulphur, the addition of potassium permanganate (1%) or nitric acid (0.25%) greatly increases the effectiveness of the fungicide, possibly owing to the formation of pentathionic acid. Independent stimulation of growth was also observed.

A. A. ELDRIDGE.

Organisms which decompose cellulose in arable land. S. A. WAKSMAN and R. J. DUBOS (Compt. rend., 1927, 185, 1226—1228).—The presence of available nitro-

gen in soils is favourable to the rapid decomposition of cellulose. The following organisms are the chief agents of this decomposition under varying conditions: (a) in saturated soils: sporogen and asporogen bacilli; (b) in aerated, acid soils: fungi; (c) in aerated neutral or basic soils: fungi and bacteria, and (in dry soil) actinomyces. *Spirochaeta cytophaga* and a similar but coccoidal form of bacteria are common in the last type of soils, and feed exclusively on cellulose.

B. W. ANDERSON.

Wyoming forage plants and their chemical composition. VII. Effect of altitude, seasonal variation, and shading experiments. E. N. ROBERTS (Wyoming Agric. Exp. Sta. Bull., 1926, No. 146, 35—89).

Soil acidity in relation to spinach production. H. H. ZIMMERLEY (Virginia Agric. Exp. Sta. Bull., 1926, No. 57, 501—521).

Mechanism of buffer action in soils. P. B. MYERS and G. L. BAKER (Delaware Agric. Exp. Sta. Bull., 1925, No. 141, 13—14).

PATENTS.

Preparation of insecticides and fungicides. CALIFORNIA FRUIT GROWERS' EXCHANGE (B.P. 267,878, 5.10.26. U.S., 17.3.26).—Insecticides and fungicides are prepared by thorough agitation of mineral oil with an aqueous solution of pectin so as to form a stable emulsion of low viscosity. [Stat. ref. to B.P. 221,466.]

C. T. GIMMINGHAM.

Adhesive compounds for combating insect pests. V. SCHOLZ and B. EIBES (B.P. 281,068, 1.11.26).—Sticky preparations for use as insect or caterpillar limes are made by adding 3—9 pts. of castor oil to a solution of a cellulose ester (e.g., celluloid waste, decamphorated or not) in 2 pts. of ethyl acetate or acetone and evaporating the solvent. In place of castor oil unsaturated oils made spirit-soluble by oxidation (e.g., linoxyn) may be used. Scents, colouring matters, and fillers may be added.

C. HOLLINS.

Manufacture from cyanamide of manures containing soluble organic nitrogen. J. BRESLAUER and C. GOUDET, Assrs. to Soc. d'ÉTUDES CHIM. POUR L'IND. (U.S.P. 1,652,753, 13.12.27. Appl., 12.5.23. Switz., 15.5.22).—See B.P. 197,690; B., 1923, 1144 A.

XVII.—SUGARS; STARCHES; GUMS.

Presence of starch in Uba cane juice. E. HADDON (South African Sugar J., 1927, 10, 629—631).—Difficult boiling of a massecuite was traced to the presence of dextrin due to starch in the Uba cane (crushed), though in other varieties this carbohydrate is absent provided the plant is ripe. Starch was identified in most of the unrefined sugars produced in South Africa.

J. P. OGILVIE.

Judging the quality of refined and direct consumption sugars. H. LUNDÉN (Centr. Zuckerind., 1926, 34, 1017—1018).—A better differentiation of the various qualities of white sugars is possible by comparison in ultra-violet light rather than in daylight. After-taste impressions, using a 2% solution, are strongly marked in the case of some qualities of unrefined white sugars. Another method of examination is to observe

the colour developed on heating the dry sugar at 100—170°, when a "first-class refined" sugar develops a straw-yellow and washed crystals a reddish-brown colour. The taste of the solutions of such heated sugars is also said to be characteristic of the different grades.

J. P. OGILVIE.

Decolorisation of [sugar] juices by activated carbons. J. WIESNER (Z. Zuckerind. Czechoslov., 1927, 52, 101—108, 120—123).—In sugar-houses producing a high-quality granulated sugar direct from juice with the use of animal charcoal, an increase in the output without a decrease of quality can be obtained with the same plant by the addition of 0.025% by wt. of the sugar present of decolorising carbon directly to the juice. This procedure is said to have a decolorising effect which is greater than can be realised by the application of the char plus carbon separately applied, less molasses being also produced, and in consequence a greater yield. It is also more economical in respect of labour and plant compared with the usual char work. J. P. OGILVIE.

Determination of invert sugar in [beet] molasses. R. OFNER (Z. Zuckerind. Czechoslov., 1927, 52, 108—112).—Previous to the determination of invert sugar, the solution of beet molasses is clarified with lead acetate, and the excess of this reagent removed; the filtrate is then treated with about 1 g. of good activated carbon (e.g., "norit") per 100 c.c. of a 20% solution, in order to remove nitrogenous copper-reducing compounds. Fehling's solution, the alkalinity of which has been largely reduced by the addition of sodium phosphate solution, is employed.

J. P. OGILVIE.

Dust fungicides. LEE and MARTIN.—See XVI.

PATENTS.

Purification of sugar solution. ELEKTRO-OSMOSE A.-G. (GRAF SCHWERIN GES.) (B.P. 272,943, 16.6.27. Ger., 16.6.26).—A modification of G.P. 361,594 (B., 1923, 369 A) in which the sugar solution is subjected to the action of a continuous electric current, the electro-positive diaphragm originally before the anode being replaced, except in the last cell of a series, by one of electronegative character similar to that before the cathode, whilst the water which flows through the cathode chamber, where it develops an alkaline reaction, is afterwards made to flow through the anode chamber, so that the sugar solution is prevented from becoming acid.

F. R. ENNOS.

Treatment of sacchariferous [sugar] solutions. O. S. OLSEN, Assr. to W. F. CLEGG (U.S.P. 1,652,680, 13.12.27. Appl., 14.6.26).—A syrupy mixture containing crystals, by flowing into a sugar-purging centrifugal, has a depressed surface, and wash-water is discharged into the depression in order to dilute the syrup present in the mixture.

B. FULLMAN.

Manufacture of dextrose. C. COPLAND, Assr. to INTERNAT. PATENTS DEVELOPMENT CO. (U.S.P. 1,652,393, 13.12.27. Appl., 30.11.25).—Dextrose is crystallised by mixing with a magma of dextrose solution and crystals a mother-liquor obtained from another mass of crystals and subjecting the mixture to a crystallising operation while maintaining the crystals dispersed and in motion through the liquid. Crystals and liquor are finally separated.

C. O. HARVEY.

Desugaring of molasses. G. L. SPENCER, Assr. to CUBAN-AMERICAN SUGAR CO. (U.S.P. 1,646,323, 18.10.27. Appl., 31.10.23).—The molasses is diluted with water and treated with tribasic barium silicate to precipitate the sucrose as barium saccharate and part of the barium as monobasic silicate. After collecting the precipitate on a filter it is washed with barium hydroxide solution and treated with sulphurous acid in sufficient quantity almost to saturate the barium in the saccharate, whereby the sucrose is set free with precipitation of barium sulphite. The sucrose is obtained in a state of purity by evaporation of the filtrate, whilst the mixture of barium sulphite and monosilicate is calcined at about 1425—1500° with formation of tribasic barium silicate and sulphur dioxide, both of which are returnable to the process with slight loss.

F. R. ENNOS.

XVIII.—FERMENTATION INDUSTRIES.

Rôle of buffer substances in fermentation industries. R. LOMBAERS (Chim. et Ind., 1927, 18, 586—590).—A theoretical discussion.

Variations of free acidity and the concentration of buffer substances during brewing. F. DUFOUR (Chim. et Ind., 1927, 18, 594—599).—Except for a slight increase of free acidity on the boiling of the mash, the p_H varies little during mashing. The increase is more marked if raw grain is used partly to substitute the malt. On boiling the wort with hops the increase of free acidity is greater. Raw grain contributes a negligible quantity of buffer substances to the wort. The majority of buffer substances are pre-formed in the malt and, being directly soluble, enter more easily into solution the greater the dilution. The boiling of the mash diminishes the amount of buffer substances, the elimination being proportional to their original concentration. Although a certain quantity of buffer substances is precipitated and removed on boiling the wort with hops, their concentration alters little owing to the hops contributing approximately an equivalent amount to the solution. The extraction of buffer substances by sparging is relatively greater than that of the other substances. With worts of low gravity, the concentration of buffer substances is less and the buffer index higher than with worts of strong beers. Although the boiling of the mash assists in preventing an excessive quantity of buffer substances in the wort, the chief control of their concentration lies in the method of malting.

C. RANKEN.

Titration curves of wort and beer. M. VAN LAER (Chim. et Ind., 1927, 9, 591—594).—If the values of p_H , which are obtained by adding quantities of acid or base to the liquid under examination, are plotted against those additions, the titration curve is the result. The curve makes with the p_H axis an angle, α , which is proportional to the concentration of the buffer substances in solution. An abrupt change of the direction of the curve indicates the disappearance of the buffer capacity at that point. The buffer index is calculated from the formula $10/(p-p_1)E$, where p is the p_H of the original wort, p_1 that of the wort with 1 c.c. of 0.1N-acid added to 25 c.c. of the wort, and E the extract in degrees Balling. Van Slyke put forward the alterna-

tive formula of $\pi = dB/dp_H$, where π was termed the buffer coefficient and B the equivalents of base per litre. For liquids containing several buffer substances in solution this formula is more suitable. If the separate buffer coefficients are, respectively, $\pi_1, \pi_2, \pi_3 \dots$, the total buffer effect will be $\Sigma\pi = \pi_1 + \pi_2 + \pi_3 + \dots$. From these data the p_H corresponding to each total buffer coefficient can be calculated, and a curve traced showing a series of maxima and minima. The curve makes evident the low buffer capacity of barley and that the characteristic curves for wort and beer appear only after malting. The titration curve changes slightly in passing from wort to beer, a slight increase of buffer showing in the region of organic acids and a slight diminution in the phosphate zone. Three maxima characterise the beer curve, the first at p_H 4.2, corresponding to the maximum buffer capacity of succinic acid; the second at p_H 6.9, corresponding to the maximum buffer capacity of the primary phosphate; and a third towards p_H 8.6. In some cases a maximum may also occur at p_H 6.5 due to carbonic acid. Minima occur at values of 5.8 and 7.6, and as the p_H value of the wort before fermentation is generally less than the former of these values, the yeast during fermentation has to render acid a liquid in which the amount of buffering gradually increases. C. RANKEN.

Musts and wines from grapes attacked by Eudemis. J. H. FABRE and E. BRÉMOND (Ann. Falsif., 1927, 20, 524—535; cf. Hughes, B., 1921, 557A).—These musts and wines are abnormal in composition, the degree of abnormality depending on the extent to which the grapes are attacked. The musts have a higher acidity than those from sound grapes, and the alcoholic content of the wines which they yield cannot be calculated with accuracy either from the density of the musts, owing to the presence of larger amounts of pectinous materials, proteins, and gums, or from the proportion of reducing substances, on account of the formation of non-volatile organic acids, especially lactic acid, from some of the fermentable sugar. Compared with those prepared from the corresponding sound grapes, the wines show an increase in density (often above 1000), dry extract, and acidity, both volatile and non-volatile, but a slightly lower alcoholic content. They are also deeper in colour, of rather a bitter taste, which improves on ageing, and do not conform to the ordinary tests for the genuineness of normal wines. F. R. ENNOS.

Wines of Morocco. L. CHAUVEAU and A. VASSEUR (Ann. Falsif., 1927, 20, 510—524).—A description of the climate, methods of culture and diseases of the grape, and quality of the wines produced in different parts of the country, together with analyses of 193 wines prepared between 1917 and 1925, and the application of the ordinary rules of oenology thereto. F. R. ENNOS.

PATENTS.

Treatment of beer wort and beer or other foaming liquids. HANSENA A.-G., and L. NATHAN (B.P. 280,395, 28.12.26).—The filtering medium, *e.g.*, asbestos, is mixed under pressure and, if desired, under sterile conditions, with a portion of the foaming liquid, and the mixture repeatedly passed through a suspension filter, so that the filtering medium is deposited upon a for-

minous or perforated support in the filter. The whole of the liquid, after removal of the heavier suspended particles by deposition or coarse filtration, is subsequently passed through these layers under approximately constant pressure. The wort, filtered under those conditions, is continuously agitated during fermentation by a current of carbon dioxide to prevent the subsidence of the yeast. C. RANKEN.

Preparation of concentrated potassium solutions from distillers' mash. R. GOLDSCHMIDT (B.P. 268,790, 30.3.27. Czechoslov., 30.3.26).—The suspended matter is centrifuged from distiller's mashes and the clean separated liquid used for preparing a new mash. The resulting mash is in turn centrifuged, and the separated liquid again used for mashing. The process is repeated until the concentration of salts in the liquor interferes with the fermentation. The potassium salts in the mash may then be worked up in the usual manner. C. RANKEN.

XIX.—FOODS.

Influence of separation and pasteurisation on size and distribution of fat globules in milk and cream. D. A. BECKETT (Econ. Proc. Roy. Dublin Soc., 1927, 2, 303—317).—The number and size of the fat globules in milk and cream were determined by the photomicrographic method of Cooper, Nuttall, and Freak, using dilutions of 1 in 5 or 1 in 20 for the milk and 1 in 105 for the cream. The percentages of fat by wt. calculated from these figures were 4.58% and 50% for the milk and cream, respectively, as compared with the values 3.4 and 47.99 obtained by the ordinary methods of extraction, the discrepancies probably being accounted for by the difficulty of obtaining a small representative sample of the diluted material. Calculations were also made of the total volumes of fat globules of various diameters from 1μ or over expressed as a percentage of the total volume of globules of all sizes present, and curves drawn expressing the relation between the percentage volume and diameter. For fresh creamery milk the maximum percentage volume (27.8) was shown by globules of 5μ diameter, the average volume of the globules being $28\mu^3$, and no marked change of distribution in globule size was shown on heating the milk. Whilst both separated and pasteurised cream showed a distribution of the smaller globules similar to that in milk, there was an increase in the number and maximum size of the larger globules, especially in the case of the pasteurised cream, for which the average globule volume was $44\mu^3$. The increase in size of the fat globules on pasteurisation of the cream may in part account for the softness of creamery butter as compared with farm butter, which has not undergone this process. F. R. ENNOS.

Starch in flour. C. L. ALSBERG (Cereal Chem., 1927, 4, 485—491).—A review of the role of starch in flour and bread and its importance in the technology of bread-making. J. R. NICHOLLS.

Value of hydrogen-ion concentration and buffer value determination in testing and use of flours. C. G. HARREL (Cereal Chem., 1927, 4, 423—435).—The development of bread due to mechanical, chemical, and enzymic treatment is discussed, and examples are

given to show how the determination of the p_H and buffer values of flour, dough, and bread may assist in bakehouse control. J. R. NICHOLLS.

Use of chromate solutions as comparison standards for the determination of "gasoline colour values." H. JØRGENSEN (Cereal Chem., 1927, 4, 468—469).—Two experiments are detailed which show that solutions of chromates and dichromates are hydrogen-ion indicators. By varying the buffer mixture in which the chromate is dissolved it is possible to prepare solutions possessing many different *defined* red-yellow and yellow colour tones. It is suggested that such solutions might be used as comparison standards in determination of "gasoline colour values." J. R. NICHOLLS.

Determination of flour ash. J. MICKA (Cereal Chem., 1927, 4, 461—467).—Three methods of ashing flour were examined, viz., raising the temperature slowly from 24° to 705°; raising the temperature rapidly from 149° to 815°; and placing it in a furnace at 926°. Partial fusion took place in the first process, but in the others fusion was complete. The times for ashing were, respectively, 2 hrs., 70 min., and 16 min. with platinum vessels, but longer with porcelain. In platinum ware, fusion of the ash did not lead to irregular or erroneous results, and such substances as the alcohol-glycerol reagent and alundum did not shorten the time of burning at high temperatures. The addition of calcium acetate effected this, but the results were unreliable unless the temperature of the furnace exceeded 900°. The best method proved to be the direct burning at high temperatures in platinum without addition of other substances. J. R. NICHOLLS.

Physical properties of washed gluten. T. R. JAMES and L. X. HUBER (Cereal Chem., 1927, 4, 449—460).—A machine designed for measuring the force necessary to stretch and break a thin layer of gluten is described. The breaking force differs with the variety of wheat, the glens from hard red spring wheats being the strongest. The gluten of a patent flour normally has a higher breaking force than that of the clear flour. Nitrogen trichloride and chlorine, as applied in commercial mill treatment, considerably increase the breaking force. Fine grinding with a cool grinder had no effect, but high temperatures in air for an extended period of time increased the breaking force of the gluten. Chlorine and hydrogen chloride affect the breaking force of the gluten in somewhat the same way, but a comparison of the effects on the same flour shows distinct differences. Glutens with high breaking force are needed for good baking strength, but such glutens may fail through other causes. J. R. NICHOLLS.

Heat coagulation of gluten. C. L. ALSBERG and E. P. GRIFFING (Cereal Chem., 1927, 4, 411—423).—An attempt has been made to ascertain the temperature of coagulation of gluten. An indirect method was devised based on the assumption that coagulated gluten swells less in weak acid. The swelling power ("coefficient of imbibition") was measured by the ratio of the weight of a disc of gluten after swelling in weak acetic acid to its original weight. Heating glutens to temperatures between 50° and 80° caused a decrease in swelling power, the decrease for 1° being approximately constant

throughout this range except between 60° and 65°, when it was greater. There was, however, no indication of a definite coagulation point. When the gluten was heated at temperatures between 30° and 50° there was no impairment of swelling power; a slight increase was even indicated. J. R. NICHOLLS.

Chemical changes incident to ripening and storage in the Grimes apple. F. GERHARDT (Plant Physiol., 1926, 1, 251—264).—The ripening process is associated with loss of moisture, acidity, dextrins, starch, and acid-hydrolysable material; the density, sugars, and soluble pectin increase. There is little correlation between chemical composition and keeping quality of cold-storage apples. CHEMICAL ABSTRACTS.

Fruit jellies. P. B. MYERS and G. L. BAKER (Delaware Agric. Exp. Sta. Bull., 1926, No. 141, 14—19; No. 144, 3—35).—The minimum point of jelly formation is p_H 3.50—3.55, regardless of total acidity or the presence of salts; the optimum point is p_H 2.85—3.30, depending on the nature of the acid and salt used. The effects on jelly formation of variations in the hydrogen-ion and salt concentrations were studied. CHEMICAL ABSTRACTS.

Apple juice in "pure fruit" conserves (cherries and strawberries). C. F. MUTTELET (Ann. Falsif., 1927, 20, 535—537).—Cherries contain a slight excess of dextrose over laevulose varying from 0.7 to 1.5%, but strawberries contain the two sugars in very nearly equal amounts, an excess of dextrose being found in some cases up to 0.2%. The adulteration of conserves made from these fruits with filtered apple juice may therefore be detected in the same manner as with other fruits (cf., B., 1927, 122, 502) by the presence of an excess of the laevorotatory sugar. Two household conserves of these fruits showed an excess of dextrose in agreement with the figures for the pure fruit, but some of the commercial preparations examined contained a decided excess of laevulose over dextrose (1.6% and 0.7% for the cherry and the strawberry conserves respectively), and were obviously adulterated with apple juice, confirmation of this being afforded by the presence of malic acid (cf. B., 1922, 726 A). F. R. ENNOS.

Influence of method of preparation on chemical composition of preserved peas. LASAUSSE (Ann. Falsif., 1927, 20, 539—542).—Two methods are used for preserving peas. In one the peas, after cooking, are washed with plenty of cold water before packing, and in the other they are merely drained without cooling. Peas which had been gathered at four different periods during the season and sieved to three grades were analysed both in the fresh and preserved condition. The results showed that as the season advanced there was in peas of the same grade a gradual increase in dry extract, starch, and total nitrogen, these values being highest for the fresh peas and lowest for the preserved. F. R. ENNOS.

Biological evaluation of food preparations. E. WALDSCHMIDT-LEITZ (Z. Unters. Lebensm., 1927, 54, 291—294).—A classified analysis is made of the amino-acid contents of a food preparation known as "Promonta," showing arginine 0.78%, lysine 0.80%, histidine

0.64%, tryptophan 0.14%, tyrosine 0.99%, and cystine 0.74% of total dry matter. A. G. POLLARD.

Protective tubes for thermocouples for determining heat penetration in processed foods. K. L. FORD and A. G. OSBORNE (Ind. Eng. Chem., 1927, 19, 1345—1346).—Bakelite makes an ideal substitute for glass or metal tubes for thermocouples, as it possesses a lower heat conductivity than metal and a greater resistance to failure than glass. When the thermocouples are used for heat-penetration determinations, and their ends are exposed to the heating medium, the accuracy of the readings is affected by the heat conductivity of the protective tube, the error depending on the rate of temperature change and on the nature of the material being tested. F. R. ENNOS.

PATENTS.

Treatment of cereals. L. BARTMANN (B.P. 280,605, 18.5.26).—The grain is steeped in water at 30—35° whereby the endosperm swells and becomes converted into a soft pasty mass, which is expelled by mechanical means from the toughened husks. During and, if desired, after the steeping process, germination, fermentation, putrefaction, and premature action of enzymes are prevented, without injury to the nutritive matter in the grain, by means of formaldehyde gas or its aqueous solution, or by subjecting the grain to rapid changes of temperature or to electrical or mechanical shocks.

F. R. ENNOS.

Maturing of flour. J. I. LOGAN, ASSR. to INDUSTRIAL APPLIANCE CO. (U.S.P. 1,640,199, 23.8.27. Appl., 3.8.25).—Aqueous ammonia in a dissolution tank passes into a reaction tower where a mixture of chlorine and nitrosyl chloride (in excess of the amount which will combine with the ammonia), diluted with air, is forced through it from the bottom. The gaseous product is then brought into contact with the flour in a dispersed state in the treating chamber.

F. R. ENNOS.

Manufacture of bread, biscuits, cakes, and other foodstuffs. S. BONSOR (B.P. 280,729, 16.12.26).—The dough ingredients are subjected during mixing to the action of ultra-violet rays for a suitable period, *e.g.*, 5—10 min., in order to render the final product lighter and more easily digested.

F. R. ENNOS.

Sterilising clear and turbid liquids [foods] by means of ultra-violet rays. E. O. SCHEIDT (B.P. 257,956, 6.9.26).—A high-tension electric discharge is passed through the rarer gases, *e.g.*, neon and argon, contained in a series of quartz tubes which may be either U-shaped or formed of two co-axial vertical limbs one enclosing the other, and the ultra-violet rays produced are allowed to act on the liquid to be treated, *e.g.*, milk, as it flows in a thin film between the surfaces of the quartz tubes and of surrounding tubes of porcelain.

F. R. ENNOS.

Manufacture of food products. C. HOFFMAN, ASSR. to WARD BAKING CO. (U.S.P. 1,640,193, 23.8.27. Appl., 20.8.24).—A palatable, easily digested food product, rich in water-soluble vitamin-B, is made from cereal germs by extracting the oil therefrom, cooking the raw material to gelatinise the starch content, and

malting the material in the presence of sufficient acid to give the ultimate product a p_H value of 5.4—5.7.

F. R. ENNOS.

Food product. J. J. REYNOLDS (U.S.P. 1,647,749, 1.11.27. Appl., 8.11.24).—The foodstuff, *e.g.*, cheese, meats, or vegetables, is treated with an invisible, odourless, tasteless mineral oil which covers the foodstuff as a thin film at temperatures between 0° and 38°, and, by closing the pores, stops any moulding and protects it against further moulding.

F. R. ENNOS.

Manufacture of bread. H. A. KOHMAN, ASSR. to FLEISCHMANN CO. (U.S.P. 1,652,828, 13.12.27. Appl., 29.6.26).—See B.P. 244,489; B., 1927, 376.

Fine grinding of corn. R. SCHERMANN (B.P. 261,753, 17.11.26).

Travelling [bakers'] ovens of the flat-sole type. J. H. N. PELKMAN (B.P. 280,622, 16.8.26).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Explosions occurring during work with ether containing peroxide. L. BRANDT (Chem.-Ztg., 1927, 51, 981—983).—During the drying of the fatty acid extracted from soap by means of ether a violent explosion occurred due to the presence of peroxide in the extract. This may have been derived from a per-salt in the soap or have been present in the ether originally. The removal of peroxide from ether is discussed at some length; anhydrous ether may be purified by distillation over sodium and damp ether by shaking with acid ferrous chloride followed by distillation. To avoid distillation the addition of lead sulphide, precipitated by hydrogen sulphide from lead acetate solution and dried at 100°, is recommended. The use of catalysts such as platinum sponge, amalgamated aluminium, zinc-copper couple, manganese or lead dioxide, or solid ferrous sulphate is not advisable, as large volumes of oxygen saturated with ether vapour are evolved. Ether purified from peroxide rapidly acquires further quantities after exposure to the air, and should therefore be kept in dark, well-stoppered bottles containing lead sulphide. Titanic sulphate forms the best test for the presence of peroxide in ether, and the iodide method is recommended as a means of determination.

A. R. POWELL.

Anthranilic esters. A. WAGNER (Chem.-Ztg., 1927, 51, 855).—Verley's statement (A., 1927, 856) that geranyl anthranilate can only be prepared by heating methyl anthranilate and geraniol with the ethoxide of sodium, aluminium, or magnesium, is incorrect. The author has obtained 1.2 kg. of the geranyl ester from 400 g. of anthranilic acid and 1.133 kg. of geraniol. The terpinyl ester, which has an odour of lilies of the valley, is obtained in similar yield from anthranilic acid and terpineol. A mixture of methyl and geranyl anthranilates is obtained from anthranilic acid, methyl alcohol, and geraniol. Other anthranilic esters are similarly prepared. By treating the above with nitro-compounds other sweet-scented esters are obtained.

W. T. K. BRAUNHOLTZ.

Essential oils in desert plants. I. Physical constants. M. ADAMS and R. BILLINGHURST (J. Amer. Chem. Soc., 1927, 49, 2895—2897).—The following oils were

obtained by steam distillation of the plants mentioned. The percentage of oil in the plant is recorded in brackets; all b.p. are those at which the oil boiled freely under the local barometric pressure (642—647 mm.). *Artemisia tridentata typica* (0.92%), b.p. 173.5°, d^{18}_4 0.9052, $[\alpha]^{20}_D$ -8.54°, n^{20}_D 1.4689, iodine value (Hübl) 168.0; *Gutierrezia sarothrae* (0.33%), b.p. 157.5°, d^{18}_4 0.8678, $[\alpha]^{20}_D$ +36.49°, n^{20}_D 1.4741, iodine value 304.4; *Juniperus utahensis*, b.p. 163.5°, d^{18}_4 0.8879, $[\alpha]^{20}_D$ +56.36°, n^{20}_D 1.4715, iodine value 224.7; *Salvia lanceolata* (0.32%), b.p. 185.5°, d^{18}_4 0.9254, $[\alpha]^{20}_D$ +2.35°, n^{20}_D 1.4771, iodine value 110.9; *Thamnosma teranum* (0.05%), b.p. 128°, d^{18}_4 0.9593, $[\alpha]^{20}_D$ +57.67°, n^{20}_D 1.4737, iodine value 119.4; *Tetrademia glabrata* (0.12%) b.p. 163°, d^{18}_4 0.8522, n^{20}_D 1.4756, iodine value 189.6; *Chrysothamnus graveolens* (0.85%), b.p. 169.5°, d^{18}_4 0.8746, $[\alpha]^{20}_D$ -1.62°, n^{20}_D 1.4842, iodine value 184.1; *C. viscidiflorens elegans*, b.p. 162°, d^{18}_4 0.8549, $[\alpha]^{20}_D$ +20.67°, n^{20}_D 1.4797, iodine value 325.3; *C. nauseosus graphalodes* (0.49%), b.p. 159°, d^{18}_4 0.8651, n^{20}_D 1.5080; *C. naus. viridulens* (0.17%), d^{18}_4 0.9045, n^{20}_D 1.5008; *C. naus. hololeucus* (0.18%), b.p. 160.5°, d^{18}_4 0.9767, n^{20}_D 1.5399; and *C. naus. Mohavensis* (0.16%), b.p. 126°, d^{18}_4 0.8924, n^{20}_D 1.4971.

F. G. WILLSON.

PATENTS.

Manufacture of alkamine esters of N-substituted o-aminobenzoic acids. I. G. FARBERIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (B.P. 260,605, 28.10.26. Ger., 28.10.25. Addn. to B.P. 241,767; B., 1926, 107).—The alkamine esters of anthranilic acids carrying on the nitrogen atom alkyl or alkoxyalkyl groups with more than two carbon atoms are equal as local anaesthetics to the corresponding p-aminobenzoic esters of the prior patent, and superior to cocaine. o-β-Methoxyethylaminobenzoic acid, m.p. 102°, obtained from sodium anthranilate and methyl β-chloroethyl ether, gives an ethyl ester, b.p. 160—165°/8 mm., which, when boiled with 1-β-hydroxyethylpiperidine and a little sodium ethoxide, is converted into β-1-piperidinoethyl o-β-methoxyethylaminobenzoate (monohydrochloride, m.p. 118°). C. HOLLINS.

Preparation of measles toxin and antitoxin. PARKE DAVIS & Co., Assees. of N. S. FERRY (B.P. 267,520, 8.3.27. U.S., 15.3.26).—From the blood of a patient in the early stages of measles (*Morbilli*), on incubation in a suitable medium, there is isolated a micrococcus (usually a streptococcus, e.g., *Streptococcus morbilli*, but sometimes appearing as a diplococcus) specific to measles. From this, on cultivation in 0.2% glucose broth, there is obtained a corresponding toxin. This may be used for the identification of the measles organism, for determining susceptibility to measles, or for preventive inoculation. For the production of measles antitoxin the toxin (sterile or otherwise, as desired) is injected into animals, preferably into horses, the animal bled, and the serum or plasma concentrated and refined.

B. FULLMAN.

Obtaining highly active substances from female internal secretory organs. SOC. CHEM. IND. IN BASLE (GES. F. CHEM. IND. IN BASEL) (B.P. 265,567, 14.1.27. Switz., 2.2.26).—Extracts from female internal secretory organs or distillates from the extracts are hydrolysed by

means of alkali in absence of oxygen. E.g., extract of ovary, purified as in B.P. 226,372 (B., 1925, 149), is treated in warm alcoholic solution with alcoholic potash to alkalinity, in a hydrogen atmosphere. On neutralisation with carbon dioxide, evaporation *in vacuo*, extraction with acetone, etc. the active product is obtained as a bright oil.

B. FULLMAN.

Iodine-substituted benzonitriles (B.P. 275,213).—See III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Preservation of photographic materials. REPORT OF THE COMMITTEE (Phot. J., 1927, 67, 498—499).—No recommendations can be made which will ensure permanence of photographic records for all time, and especially are records made on celluloid and cellulose acetate not likely to be permanent for long periods of years. The following recommendations are made for the preservation of existing records on glass, paper, and celluloid: Negatives and positives on glass should be soaked in water, refixed, washed, hardened in chrome alum, washed again, dried thoroughly, and then varnished with a shellac varnish; those on celluloid (e.g., roll and flat films) should be treated as for glass, but there seems to be no suitable varnish for celluloid films. Kinematograph films should be well dried in warm air before packing. In the case of bromide prints, sulphide toning gives permanence at least equal to any other method of production of the prints. Finished silver prints, not toned, should be soaked, refixed, washed, sulphide toned, rewashed, hardened in chrome alum, washed again, and dried. If mounted, dry-mounting must be used. New prints should be made on a paper in which the emulsion is coated on a non-baryta-coated, pure rag paper. Mounted finished prints should be copied and fresh prints made. All prints should be thoroughly dried and varnished with gum dammar in benzene. In storing, prints should be interleaved with a pure raw photographic base paper packed in two wrappings of wax paper, and kept in a cool dry place to which noxious fumes do not have access. A formula for a sulphide toning bath is given, as well as tests for thiosulphate and for silver in determining the thoroughness of fixation. W. CLARK.

Photographic development in bright light. LÜPPO-CRAMER (Z. angew. Chem., 1927, 40, 1225—1231).—A historical summary and brief review of the present position of desensitisers enabling colour-sensitive films to be developed in bright light. The disadvantage of phenosafranine, viz., its staining action on hands and films, is overcome in the more recent desensitisers Pinakryptol Green and Pinakryptol Yellow, which are substituted flavindulines, the simplest representative being prepared by condensing o-aminodiphenylamine with 2-aminophenanthrenequinone. The desensitising action is a chemical one, and is ascribed to the oxidising action of the dyes used, their leuco-derivatives possessing no desensitising properties. W. T. K. BRAUNHOLTZ.

Tentative hypothesis of the latent image. A. P. H. TRIVELLI (J. Franklin Inst., 1927, 204, 649—662, and Phot. J., 1928, 68, 14—20).—Consideration of the

facts that some substances, including the silver halides and silver sulphide, are photo-conductive as well as photo-electric, that in the silver halide grain, silver sulphide and probably silver exist, and that these appear to be more photo-electric than the surrounding silver halide, leads to the view that the photo-electric effect and photo-conductivity may play a part in the formation of the latent image. It is assumed that when light falls on the silver halide grain containing sensitising specks of silver or of silver sulphide, the greater photo-electric effect of these speck substances compared with that of the silver halide produces a potential difference, and that in some way not known the photo-conductivity produced by the light action causes an electrolytic deposition of silver in the neighbourhood of the speck, so increasing its size and in consequence the developability of the grain. Such an effect would not preclude the simple photochemical decomposition of the silver halide by light to give silver, nor the possibility of silver sulphide acting as a halogen absorber. Qualitatively there seems to be a correlation between the intermittency effect and also the failure of the reciprocity law, and the latent image formation. Halogen absorbers which prevent solarisation accelerate the Herschel effect.

W. CLARK.

Preparation and properties of some synthetic photohalide emulsions. E. P. WHITEMAN, A. P. H. TRIVELLI, and S. E. SHEPPARD (Phot. J., 1927, 67, 500—504).—See B., 1927, 925.

PATENTS.

Colour photography. M. MARTINEZ (B.P., 280,053, 11.5.26).—A compound-negative sensitive material, designed to produce the three-colour separation negatives in a single exposure, is formed of superimposed layers in non-adhesive contact with each other, having coatings of successively increasing speeds and different colour sensitiveness, the slowest layer being used towards the lens, without requiring colour filters or special colouring of the coatings. The back emulsion of highest speed is red-sensitive, the middle orthochromatic, and the slowest, front emulsion is an ordinary one, chiefly sensitive to the blue and violet. The opacity of the emulsions increases from the front to the back. The coatings for the negatives are such as to give black and white images on development, whilst the coatings for the positives to be printed from these negatives are such as to give, directly on exposure, images in colours complementary to those for which the corresponding negative sections were sensitised (cf. B.P. 280,252, following). After printing, the positive sections are washed and superimposed.

W. CLARK.

Manufacture of photo-sensitive surfaces [for use in colour photography]. M. MARTINEZ (B.P. 280,252, 17.5.26 and 7.1.27).—In the three-colour process described in B.P. 280,053 (preceding), the red, blue, and yellow images to be superimposed are prepared as follows: for positives to print in red from a black and white negative, the sensitiser consists of alloxan or another derivative of the urea group of the same type, such as alloxanic acid, alloxantin, or murexide, either alone or in conjunction with each other or with a suitable metal salt or salts, such as alkali or other oxalates,

ferric or mercuric salts. The sensitiser to print blue consists of two solutions; the one has as its basis ferric salts in conjunction with a cyanide, *e.g.*, mercuric or potassium cyanide; the other potassium ferriocyanide. The sensitiser to print yellow has as its basis silver lactate in conjunction with metal oxalates and ferric salts, preferably also with an excess of lactic acid. After printing, the surfaces require treatment with water only.

W. CLARK.

Manufacture of photographic plates or films. I. G. FARBERIND. A.-G. (B.P. 275,933, 4.5.27. Ger., 12.8.26).—An anti-halation layer for use between the emulsion and the support is made from a solution of an organic hydrophilic colloid (*e.g.*, gelatin) containing manganese compounds and treated by electro-dialysis in the middle compartment of a three-cell apparatus having an electro-positive diaphragm between the anode and middle cells and an electro-negative diaphragm between the middle and cathode cells.

W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

Influence of coefficient of exchange on ease of saturation of guncotton. I, II. P. VIEILLE (Mém. Poudres, 1927, 22, 307—310, 311—314).—I. The "coefficient of exchange" during washing has been determined for C.P. 1 (guncotton) when saturated with calcium carbonate. Even if a low coefficient does not of itself imply a successful stability test at 110°, it appears to be necessary after a short carbonatation. A mere boiling of C.P. 1 in waters kept alkaline will not enable a guncotton to show high stability tests even if the coefficient of exchange has been brought to a moderately low value. The process of purification of guncotton is almost independent of the strength of baths with an alkalinity of 0.01 g./litre.

II. A washing maintained for 150 hrs. does not alter the coefficient of exchange from that obtained after 80 hrs. Prolonged acid-washing neither increases the solubility nor causes denitration. An exchange coefficient of 0.014 is insufficient to give good results in the 110° stability test after carbonatation, and an 80 hrs. boil appears to be necessary for a successful carbonatation.

S. BINNING.

Effect of temperature on efficiency of washing a guncotton. P. VIEILLE (Mém. Poudres, 1927, 22, 315—316).—The influence of temperature on the washing of C.P. 1 has been investigated on a previously decarbonated sample. Coefficients of exchange were determined at temperatures ranging from 89.51° to 10°. The coefficients vary with the temperature at the same rate as is generally accepted for many chemical reactions.

S. BINNING.

Purification of guncotton. P. VIEILLE (Mém. Poudres, 1927, 22, 317—319).—Increase of acidity in the bath during purification of guncotton has been investigated. Of the total increase, 84% is due to nitric acid, 8% to sulphuric acid, and 8% to various unspecified organic acids.

S. BINNING.

Inflammability of nitro-cottons. I, II. P. PASCAL (Mém. Poudres, 1927, 22, 285—298, 299—306).—I. The relation between moisture content and inflammability, either superficially or throughout the mass, of various

types of nitro-cotton has been investigated. The inflammability remains much the same for all types of nitro-cotton, with the same moisture content, at all stages of manufacture before beating. After beating, the inflammability decreases. C.P. 1 prepared by the displacement process is much easier to ignite than C.P. 1 made by the pot process, and the same relation holds good after beating. C.P. 2 has about the same inflammability as pot C.P. 1.

II. C.P. 1 made in Selwig nitrators at Moulin Rouge factory is as inflammable at all stages of the process as displacement C.P. 2 from Angoulême, but C.P. 2 from Angoulême is notably less inflammable than that from Moulin Rouge. The inflammability appears to increase with the percentage of nitric acid in the nitrating bath. These experiments justify the regulation that during transport nitro-cottons should have a moisture content of 30%, which ensures a relative safety if the density of the packed charge is 0.3 as is usually the case.

S. BINNING.

Coefficients of exchange of nitro-cotton. A. KOEHLER and M. MARQUEYROL (Mém. Poudres, 1927, 22, 320—325).—An improved method of determining the coefficient of exchange in the stabilisation of nitro-cotton is described, which enables this value to be determined at 100° instead of at 130° as in the regulation method, and does not require the use of an autoclave. Relations between the coefficients at 130°, 100°, 90°, 75°, 60°, and 40° are tabulated. S. BINNING.

Determination of oxalates in nitroglycerin powders. M. TONEGUTTI (Annali Chim. Appl., 1927, 17, 531—534).—Determination of the oxalate added to nitroglycerin powders by extraction with dilute acid and titration with permanganate gives low results, although determination of the amount of the cation of the oxalate gives almost normal values. It is concluded that part of the added oxalate undergoes some transformation in the powder. T. H. POPE.

Determination of water in glycerin for [the manufacture of] dynamite. T. BERTH (Chem.-Ztg., 1927, 51, 975—976).—Exactly 100 g. of the glycerin are mixed with 150 c.c. of tetrachloroethane and 2.5 g. of finely-powdered silver nitrate in the distillation flask of a Tausz and Rumm apparatus (B., 1926, 338). The mixture is distilled over a small flame until no further drops of liquid are seen in the bulb tube. The water layer is withdrawn into a capillary measuring tube previously wetted with tetrachloroethane and its volume read to 0.01 c.c.; the figure so obtained is the percentage of moisture in the sample. A. R. POWELL.

PATENT.

Reducing the viscosity and increasing the solubility of nitrocellulose. S. D. SHIPLEY, Assr. to ATLAS POWDER Co. (U.S.P. 1,652,587, 13.12.27. Appl., 9.6.22).—Solutions of low viscosity and high concentration are made by heating nitrocellulose in a non-solvent liquid in the presence of aluminium as catalyst. B. FULLMAN.

XXIII.—SANITATION; WATER PURIFICATION.

Hydrogen-ion control of sludge digestion. G. M. FAIR and C. L. CARLSON (Eng. News-Rec., 1927,

99, 881—883).—The digestion of properly seeded sewage solids as measured by gasification normally proceeds in three stages. There is first a short period of relatively rapid digestion during which the p_H value of the sludge decreases, and with it the rate of gasification, followed by an extended period of very slow or inhibited digestion characterised by low gas production and low but rising p_H value. During the third stage the p_H value rises to about 6.8, and methane production becomes active. The long dormant period can be eliminated and gasification greatly accelerated by adjusting the p_H value of the sludge by the addition of lime, marble dust, or dolomite dust. The period of digestion of such controlled sludge is about 8 weeks, which is about one third the time required for normal uncontrolled digestion. Experiments with respect to the digestion of sludge under different conditions of hydrogen-ion concentration, viz., p_H 6.8, 7.2, and 7.6, indicated that the optimum reaction of controlled digesting sludge varies according to the stage of digestion reached by the decomposing materials. It is probable that a p_H value slightly below the neutral point, say 6.8, is the optimum during the initial stage of digestion, and one slightly above that point, say 7.2, during the final stage. Further evidence is required before the path of optimum p_H value is definitely established; variation with the nature of the sewage is probable. W. T. LOCKETT.

Biology of sewage disposal. Chemical studies on Imhoff tanks. W. RUDOLFS and F. L. CAMPBELL (New Jersey Agric. Exp. Sta. Bull., 1926, No. 427, 103 pp.).—Variations in the carbon dioxide content of the tank gases are correlated with the resting and operating periods of the tanks. The composition and flora of the liquid between sludge and scum are a better indication of the activities in the tank than is the analysis of sludge filtrate. CHEMICAL ABSTRACTS.

Bactericidal effect of chlorine in water. A. ROMWALTER (Gas- u. Wasserfach, 1927, 70, 1133—1136).—The statement that the strong action of dilute disinfectants, particularly hypochlorite solution, on water bacteria is due to the bactericidal effect of ultra-violet rays emitted by the disinfectants is investigated. Experiments were made with hypochlorite solutions of various strengths contained in a fused quartz crucible or in an enamelled iron vessel, and water containing bacteria was brought into contact therewith in the dark, a sensitive photographic plate also being exposed to the action of the hypochlorite. In no case was any image produced on the photographic plate, and the bacteria were killed in all cases except when hypochlorite of 0.00015 g./litre was employed for 1 hr. The oxidising effect was proved by the production of nitrogen from ammonium chloride and the oxidation of ferrous sulphate. Sources of error in the original experiment of Bunau-Varilla and Téchoneyres (cf. B., 1925, 523) are discussed, the conclusion being that the bactericidal effect of chlorine is a function of the total quantity of oxidisable impurities in the water treated.

W. G. CAREY.

Elimination of silica from waters containing silicic acid. E. BERL and H. STAUDINGER (Z. angew. Chem., 1927, 40, 1313—1317).—The solubility of freshly

precipitated calcium silicate at 20° is 0.24 g./litre (14.5° of hardness), and is increased by the presence of sulphates or chlorides. Silica is eliminated by treatment with calcium hydroxide, less satisfactory results being obtained by the use of calcium sulphate or chloride. Excess of calcium hydroxide is necessary if chlorides are present, twice the theoretical amount being used if the ratio of silica to sodium chloride is 1 : 1.25. Excessive alkalinity produced by precipitating the excess of calcium hydroxide with sodium carbonate is avoided by softening with lime and soda. When silica and calcium sulphate are both present the former and the temporary hardness are removed by calcium hydroxide, and the excess of the latter together with the sulphate by treatment with sodium bicarbonate. The calcium silicate formed in the absence of chloride contains equimolecular proportions of lime and silica, but when chlorides are present less silica is precipitated per unit of lime. Compounds of the type NaHCaSiO_4 are also found.

W. G. CAREY.

Detection and determination of small quantities of manganese in water. R. SCHMIDT (Chem.-Ztg., 1927, 51, 1015—1016).—As little as 0.004 mg. of manganese may be detected in 100 c.c. of water by rendering it alkaline with sodium hydroxide, oxidising the manganese with a current of air, acidifying, and adding a solution of dimethyl-*p*-phenylenediamine; a reddish-violet colour indicates the presence of manganese. Iron interferes but not in the presence of citric acid. If the water contains nitrite this must first be destroyed by adding 2 drops of a 5% solution of sodium azide. For quantitative work 100 c.c. of the water are treated with 3 c.c. of 3*N*-hydrochloric acid and 4 c.c. of 3*N*-sodium hydroxide, a current of air is passed through for 5 min., 2 drops of a 2% solution of the reagent are added, followed by 10% citric acid solution drop by drop until the violet colour appears, and the colour is compared with that of a standard manganese solution containing approximately the same amount of iron and prepared in a similar manner. Low results are obtained if the water contains more than 50 mg./litre of magnesium salts.

A. R. POWELL.

Quantitative studies of phenols in water supply.

W. DONALDSON and R. W. FURMAN (J. Amer. Water Works' Assoc., 1927, 18, 605—620).—Results are given of experience in the detection and determination of phenols in a total of 556 samples of water derived from several sources. The method of determination of the tar acids (cf. J.S.C.I., 1920, 260 r) was as follows: 250 c.c. of the sample were distilled and three successive 50 c.c. tubes of distillate collected. To each were added 2.5 c.c. of diazotised sulphanilic acid—freshly prepared by mixing 5 vols. of sulphanilic acid solution (0.764%) with 1 vol. of dilute sulphuric acid (1 : 3), adding 5 vols. of sodium nitrite solution (0.34%), and packing in ice—and 2 c.c. of sodium hydroxide solution (8%). The colours were matched at the end of 5 min. against permanent standards prepared by diluting a suitable volume of potassium chloroplatinate solution (2 g. of salt in 100 c.c. of hydrochloric acid diluted to 1 litre with water) to 50 c.c. with water. With quantities of phenol ranging from 0.025 to 0.075 p.p.m., approximately 90% of the phenol is recoverable in

three tubes. From all available data the taste range for pure phenol in drinking water lies between 0.001 and 0.020 p.p.m. The cresols are more far-reaching in their influence on water supply than is phenol itself. The extent of taste depends on the amount and kind of phenol present and the chlorine dose.

W. T. LOCKETT.

Toxicity of lime-sulphur [insecticidal] mixture.

C. HARUKAWA (Ber. Ohara Inst. Landw. Forsch., 1927, 3, 379—404; cf. B., 1922, 834 A).—Experiments are described on the toxicity of lime-sulphur solutions to goldfish and to nymphs of a dragonfly, aquatic animals being used in order to have readily standardised conditions. Lime-sulphur was more toxic when made according to a formula in which the ratio of sulphur to quicklime was 1 : 1 than when it was 2 : 1. It is considered that the toxic action on these animals (and also on scale insects) is mainly due to the reducing action of lime-sulphur bringing about suffocation and to its corrosive action on the tissues, though other effects may be involved.

C. T. GIMLENGHAM.

Copper in water. MARCILLE.—See XII.

PATENTS.

Preventing or removing boiler scale or other like deposits.

H. KÖRPLINGER (B.P. 262,823, 13.12.26. Austr., 14.12.25).—Boiler scale etc. may be removed, or its formation prevented, by addition to the feed or boiler water of mixtures of small amounts of resins, balsams, etc. together with protective colloids for their dispersion, and of substances insoluble in water and volatile with water vapour, e.g., essential oils or their constituents, distillation products of resins etc., hydrogenated naphthalenes, wood oils, aromatic compounds, camphor, etc. Details are given of several suitable mixtures which in normal water are efficacious in quantities of 0.3—1.5 g./m.³

B. FULLMAN.

Increasing the base-exchange capacity of glauconite or of materials containing or similar to glauconite.

A. ROSENHEIM (B.P. 265,578, 27.1.27. Ger., 4.2.26).—The materials prior to their use as base-exchanging media are treated in the cold or hot state, and under pressure if desired, with solutions of salts of the alkalis and of ammonium having an alkaline reaction, or (preferably at a temperature near the b.p. of the solutions) with solutions of neutral salts. By another process the glauconite material is heated (at 150—250°) before or after treatment with an alkaline or neutral solution.

W. T. LOCKETT.

Production of a material [disinfectant] containing thymol and polymerised formaldehyde.

A. ABRAHAM (B.P. 281,530, 19.4.27).—On dissolving solid polymerides of formaldehyde in molten thymol a crystalline product having disinfectant properties etc. is obtained.

B. FULLMAN.

Insecticide. F. ZERNIK (U.S.P. 1,652,339, 13.12.27. Appl., 19.5.23. Ger., 26.5.22).—See G.P. 375,533; B., 1923, 1149 A.

Apparatus for the softening of water. UNITED WATER SOFTENERS, LTD., and E. B. HIGGINS (B.P. 281,416, 15.9.26).

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

FEBRUARY 17, 1928.

I.—GENERAL; PLANT; MACHINERY.

Grinding processes. W. OSTWALD and W. STEINBACH (Kolloid-Z., 1927, 43, 355—359).—The grinding of substances is considered from the point of view of the structure of the material to be milled. Quantitative experiments on the grinding of a number of different types of substances, carried out between $+50^{\circ}$ and -15° , showed in all cases that the degree of subdivision is greater the lower the temperature. E. S. HEDGES.

Grain composition of dusts and powders. W. BALTRUSCH (Z. angew. Chem., 1927, 40, 1450—1451).—By sedimentation in a viscous solution of celluloid in anhydrous acetone, a dust may be classified into sized particles, the viscosity of the solution being chosen so that differences of density of the particles exercise only a small effect compared with that of particle size. An apparatus and method are described by which dust analyses may be carried out. S. I. LEVY.

New viscosimeter and stagonometer. J. TRAUBE and J. MAGASANIK (Z. angew. Chem., 1927, 40, 1449—1450).—The apparatus consists of several capillary tubes of increasing diameter, one end of each being bent at nearly a right angle. The tubes are calibrated by employing in the narrowest water and dilute glycerin solution, in the next the same glycerin solution and one less dilute, and so on. Instead of employing several tubes, one tube inclined at increasing angles to the horizontal as the viscosity increases, and calibrated for each angle by the same method, may be used. The same tubes with appropriate modifications may be used to measure surface tensions by the drop method. S. I. LEVY.

Measurement of viscosity, especially of egg-white. J. JOCHIMS (Kolloid-Z., 1927, 43, 361—366).—The viscosity of a liquid which forms threads on drawing is a constant characteristic which can be measured by the length of the thread produced under standard conditions. To replace the present technical method of taking a sample of the liquid between two fingers and then moving them apart until the thread of liquid breaks, the author has adapted the principle of Tammann and Tampke (cf. A., 1927, 618), and an apparatus is described which gives reproducible results and in which a measurement takes only a few minutes. The whites of various hens' eggs give the same value. The viscosity falls rapidly with rising temperature. Addition of a few drops of potassium hydroxide or sodium chloride solutions decreases the viscosity. A similar effect, but to a less degree, is produced by addition of distilled water. E. S. HEDGES.

Automatic humidity control. W. H. APHORPE

and J. J. HEDGES (J. Sci. Instr., 1927, 4, 480—483).—A control which has proved satisfactory in maintaining a room at constant humidity is described. The principle involved is that of the ordinary hair hygrometer, the elongations or contractions of a horsehair consequent on variations in the humidity operating a system of electrical relays which govern the supply of dry or moist air. J. S. CARTER.

PATENTS.

Conducting catalytic reactions and absorptions. I. G. FARBENIND. A.-G., Assees. of C. SCHNEIDER and K. DIETRICH (G.P. 445,252, 5.10.23).—A catalyst comprising the carbonised residues of lignite after removal of dust and loose ash is claimed. The material is made into balls or briquettes for use. A. R. POWELL.

Drying and burning material in shafts. I. G. FARBENIND. A.-G., Assees. of F. WINKLER (G.P. 444,847, 7.3.24).—The material is charged on to a grate at the bottom of a shaft, and a current of hot waste gases from a furnace is passed under pressure through the mass so as to keep the particles suspended in the gas stream and thereby ensure rapid drying or burning. When the operation is finished the pressure in the shaft is utilised in ejecting the material therefrom. A. R. POWELL.

Recovery of volatile solvents by means of inert gases. R. OERTEL (G.P. 444,913, 10.7.25).—In the process of recovering volatile solvents by means of a circulating stream of inert gas, any oxygen taken up in the circuit is removed by passing the gas through a washing and filtering device containing an alkaline solution of pyrogallol in a suitable solvent. In the recovery of volatile liquids from non-circulating gas streams the oxygen filter may be conveniently arranged to prevent resinification of the absorbent medium. A. R. POWELL.

Recovery of volatile solvents by adsorption. METALLBANK U. METALLURGISCHE GES. A.-G. (F.P. 622,147, 17.9.26).—The gases containing the vapours of the volatile solvent are diluted with a sufficient quantity of inert gas previous to being brought into contact with the adsorbent material. A. R. POWELL.

Apparatus for recovering organic vapours from air. I. G. FARBENIND. A.-G. (G.P. 444,955, 25.5.22).—The gas stream is passed under pressure through a vessel containing an absorbent material arranged in thin layers on trays which may be heated or cooled as desired. A. R. POWELL.

Separation of volatile liquids with high boiling points. SOC. ÉTABL. BARBET (F.P. 620,332, 21.12.25).—Calcium chloride is dissolved in the liquid containing,

for example, acetic acid, crude alcohol, fatty acids, etc., which is then subjected to fractional distillation.

A. R. POWELL.

Separation of liquids [e.g., oil from water]. COMP. DES FREINS WESTINGHOUSE (F.P. 621,638, 17.9.26).—The apparatus comprises a centrifuge in connexion with a storage vessel into which the treated liquid passes, and which is provided with an automatic valve through which the separated liquids are periodically discharged.

A. R. POWELL.

Separating mixtures of two liquids. E. MERCK, CHEM. FABR., ASSEES. OF O. VON HEUSSLER (G.P. 445,240, 9.12.24).—The mixture is treated with a third liquid, with which it forms an azeotropic system, and the mixture is distilled under increased pressure.

A. R. POWELL.

Apparatus for the clarification of liquids. G. DUKET (F.P. 621,744, 22.1.26).—The liquid is centrifuged together with a heavy liquid the density of which is greater than that of the densest particles, so that the suspended matter is forced to the surface of the heavy liquid, from which it may readily be removed.

A. R. POWELL.

Apparatus for treating liquids with gases or for roasting pyrites. M. DREES (G.P. 444,911, 3.9.25).—The apparatus comprises a large cylindrical vessel containing a series of superimposed annular troughs disposed regularly around the walls in such a manner that the bottom of one trough dips just below the surface of the liquid in the next lower trough. The troughs are provided with corrugated bottoms carrying stirring devices, and their side walls slope outwards in smooth curves. The troughs are alternately fixed to the side walls and to a rotating central axis, so that liquid-sealed chambers are formed between one pair of troughs and the side walls, and between the next pair and the centre column alternately; during use the gas is forced from the central axis through the liquid into the outer chambers, and passes back again through more liquid to inner chambers. The apparatus is adapted to the roasting of pyrites by providing the troughs with heating flues and suitable rabbles.

A. R. POWELL.

Apparatus for treating gases with liquids. M. DREES (G.P. 444,912, 3.9.25).—The apparatus comprises a series of S- or Z-shaped concentric bands attached to a rotating central axis and sloping in such a way that liquid supplied to the lower curved portion of one band is forced by the rotation of the band upwards over the top curve and falls into the lower curve of the next band. Liquid from the outermost band falls into a reservoir whence it is returned by pipes to the innermost band, so that the process is continuous. By perforating the top curves of the bands the lower sides are wetted, thus providing additional surface for washing or absorbing the gas.

A. R. POWELL.

Apparatus for washing and purifying gases. L. MOURGEON (F.P. 621,347, 6.1.26).—An apparatus for removing suspended solids from gases comprises a conical vessel in the middle of which is a tube for the introduction of the gas. The end of the tube, which dips below the washing liquid, is provided with a conical valve having a ring-shaped aperture through which the gas

passes, thereby forcing the liquid upwards against a baffle plate.

A. R. POWELL.

Apparatus for the purification of air and gases from suspended matter. DEUTSCHE GASGLÜHLICHT-AUER-GES.M.B.H. (G.P. 444,910, 29.11.24).—The air or gas is passed through a slit which directs it at an angle on to the surface of a porous plate consisting of material which will adsorb the suspended impurities. To ensure a more perfect removal of these particles they may be given an electrical charge as they pass through the aperture of the slit.

A. R. POWELL.

Drying of gases and vapours by means of active adsorbent material. I. G. FARBENIND. A.-G. (F.P. 621,964, 24.9.26. Ger., 14.10.25).—The gases and a suitable adsorbent material, e.g., silica gel, activated alumina or bauxite, or adsorbent charcoal, are passed on the counter-current principle through a heated rotating cylinder.

A. R. POWELL.

Lubricant. I. G. FARBENIND. A.-G., ASSEES. OF W. WILKE (G.P. 445,116, 23.12.25).—Finely-divided ferric oxide obtained by the combustion of iron carbonyl is used as a lubricant, either alone or mixed with other substances. The material possesses lubricating properties equal to those of graphite.

A. B. MANNING.

Analytical centrifuge [for colloids]. T. SVEDBERG and J. B. NICHOLS (U.S.P. 1,648,369, 8.11.27. Appl., 10.9.23).—An apparatus for determining the minimum size of the dispersed particles in a colloidal solution comprises a horizontal glass tube which rotates in a horizontal plane through a vertical beam of light in a radial plane. The rate of rotation is sufficient to produce a continuous image. The speed at which the meniscus formed by the colloidal particles travels towards the outer end of the tube at a given rate of rotation is observed, and a modified form of Stokes' law is applied. Photographic determination of the variation in light absorption along the tube enables the volumes of the different sized particles of a non-uniform colloid also to be obtained.

T. S. WHEELER.

Laboratory tongs. E. H. FISHER, ASSR. to FISHER SCIENTIFIC CO. (U.S.P. 1,653,803, 27.12.27. Appl., 31.3.27).—The tongs consist of two jaws, one being flat and the other trough-shaped and having at the far end an inturned flange; the flat jaw terminates short of the flange, thus enabling a casserole handle to be held without a rotating or longitudinal movement.

H. ROYAL-DAWSON.

Carrying on catalytic reactions. F. A. CANON and C. E. ANDREWS, ASSRS. to SELDEN CO. (Re-issue 16,824, 20.12.27, of U.S.P. 1,614,185, 11.1.27).—See B., 1927, 175.

Recuperative furnace. C. STEIN, ASSR. to SOC. C. M. STEIN & CIE. (Re-issue 16,826, 20.12.27, of U.S.P. 1,350,624, 24.8.20).—See B., 1920, 662 A.

Pulverising or grinding mill. B. SCHERBAUM (U.S.P. 1,653,472, 20.12.27. Appl., 17.12.25. Ger., 24.12.24).—See B.P. 245,097; B., 1926, 424.

Centrifugal machine. M. L. SANSARICQ (U.S.P. 1,655,774, 10.1.28. Appl., 29.5.26. Cuba, 29.4.26).—See B.P. 272,047; B., 1927, 639.

Centrifugal liquid atomiser. F. WREESMANN (U.S.P. 1,655,932, 10.1.28. Appl., 19.8.26. Ger., 13.11.24).—See B.P. 269,774; B., 1927, 689.

Separation of solid and liquid materials (B.P. 279,525).—See II.

II.—FUEL; GAS; TAR; MINERAL OILS.

Coal cleaning and its relation to the cost and quality of coke. G. W. J. BRADLEY (Fuel, 1928, 7, 31–36).—Tests have been carried out on a Coppée washer with a number of South Yorkshire coals, the effect of washing on the ash distribution in the various sizes of coal, as well as their salt and sulphur content and coking power, being examined. Screening tests show that in any fraction the shale particles are generally larger than the coal particles, due to their more elongated or flatter shape. Shale which passed a $2 \times \frac{1}{2}$ in. oblong mesh was retained on a $\frac{1}{2}$ in. sq. mesh. Washing begins to be unsatisfactory for sizes below $\frac{1}{8}$ in. It is concluded that the fine slurry-making material should be withdrawn from the coking slack prior to washing, and should not be remixed with the washed slack; remixing results in a coke the ash of which is variable within wide limits. Moreover, the elimination of fines results in some gain in output and quality as well as uniformity. It is suggested that the price of coal be based on quality as determined by the removable dirt. The economic considerations involved are briefly discussed.

A. B. MANNING.

Absorption of oxygen by preheated coal. G. COLES and J. I. GRAHAM (Fuel, 1928, 7, 21–27).—Three coals of different types were heated *in vacuo* at temperatures from 200° to 500°, and the effect of the preheating on the amount of oxygen which they absorbed at 100° (after 48 and 180 hrs., respectively) was studied. In general, the oxygen absorption increased with rising preheating temperature to a maximum, and then fell eventually to a value lower than that of the fresh coal. The maximum occurred in the region 300–400°, and varied greatly in amount with the different coals. The oxygen absorptions at 50° and 100° of a number of other coals were determined similarly before and after heating *in vacuo* at 300°. The bituminous coals all show a substantial increase in oxidation after such treatment. The oxygen absorption by the anthracites appears to be speeded up in rate, but unchanged in total amount. The oxygen absorptions of the untreated coals are greater the higher the oxygen content of the coal, but for the bituminous and lignitic coals the percentage increase in oxidation due to preheating is smaller the greater the oxygen content, and for lignites may be negative. The production of carbon monoxide and dioxide during the oxidations has also been measured. Preheating in nitrogen under pressure has the same effect on the oxygen absorption as heating *in vacuo*. The bearing of the results on the liability of coals to spontaneous firing is discussed.

A. B. MANNING.

Dispersoid chemistry of peat. V. Dehydration of peat at temperatures below 100°. W. OSTWALD and A. WOLF (Kolloid-Z., 1927, 43, 336–345).—A description is given of an apparatus for the investigation of the loss of water by peat at temperatures of 100°

and below, with or without pressure. Peat gives up its water spontaneously, the more readily the nearer the temperature is to 100°. Intermittent passing of air through the peat accelerates the drying only at lower temperatures (e.g., 80°), but a continuous air current produces a better effect. Water is lost much more readily at 100° in a current of air, but the rate is not proportional to the pressure of air. The results support the view that the high-temperature treatment causes a destruction of the humus gel and a loosening of the bound water.

E. S. HEDGES.

Utilisation of cob char as carburising agent. H. L. MAXWELL (Proc. Iowa Acad. Sci., 1926, 33, 174).—The residue from the distillation of corn cobs can replace bone char in the carburising process.

CHEMICAL ABSTRACTS.

Cracked-oil gas used for coastal lighting. R. DELAPLACE (Compt. rend., 1927, 185, 1469–1472).—A new French lighthouse illuminating gas has the composition: methane, 51.87%; ethane and homologues, 17.61%; ethylene, 10.15%; propylene and homologues, 7.76%; acetylene and homologues, 11.30%; hydrogen, 1.28%. It has a higher calorific value than the gas formerly used, and possesses the advantages without the disadvantages of the Dutch and German products (purified by liquefaction and rectification) in that it is stable, has a good lighting power, and is easily compressed without elimination of the higher ethylenic hydrocarbons.

J. GRANT.

Compression of town gas. J. CHAPPUIS and A. PIGNOT (Compt. rend., 1927, 185, 1486–1488).—Curves in which the percentage of oxygen in town gas is plotted against the pressure show that at 20° the region of inflammability increases rapidly when the pressure rises from 1 to 50 kg., but above this pressure the upper limit remains almost constant. The maximum amount of oxygen which may be safely mixed with town gas under a pressure of 150–200 kg. is less than 10%. At 40° and 80° the increase in the region of inflammability is small, and slow-stage compression must be used to keep the temperature below 80°.

J. GRANT.

Determination of hydrogen in complex gaseous mixtures by absorption in colloidal palladium solution. A. E. BEET (Fuel, 1928, 7, 44–46; cf. Hempel, B., 1912, 911).—A 1–2% solution of colloidal palladium, stabilised by sodium protalbinat and containing sodium picrate, forms a convenient reagent for the determination of hydrogen in gas mixtures with the Bone-Wheeler apparatus. An additional absorption vessel is attached to the apparatus for the purpose, the mercury trough resting on an electric heater so that the reagent may be warmed to 50°. At this temperature absorption is complete in about 10–15 min. Carbon dioxide, oxygen, olefines, and carbon monoxide interfere with the determination, and must first be removed from the gas mixture by the usual methods.

A. B. MANNING.

Graphical calculation of gas combustion analyses. R. JELLER (Z. anal. Chem., 1927, 72, 249–261).—Eight examples of a graphical method of calculating the composition of gas mixtures from the combustion results, i.e., contraction in volume, volume of carbon dioxide

formed, and volume of oxygen used, are given, together with details of the procedure used in constructing the graphs.

A. R. POWELL.

Determination of tarry substances in oil products. N. TSCHERNOSHUKOV (Neftjanoe Chozjajstvo, 1927, 12, 697—698).—Sulphuric acid (*d* 1.84, 5 c.c.) and the product (5 c.c.) are vigorously shaken for 5 min.; if a clear separation is not obtained, 1—2 drops of naphthenic acid are added and the mixture, after shaking, is centrifuged for 5 min.

CHEMICAL ABSTRACTS.

Determination of paraffin in petroleum products. N. TSCHERNOSHUKOV (Neftjanoe Chozjajstvo, 1925, 9, 77—80).—The sample (10 g.) is shaken until dissolved with petroleum (b.p. 780°, 10—15 pts.); sulphuric acid (*d* 1.84, 15—20 c.c.) is added, the mixture shaken for 5 min. and kept for 1 hr. The volume of the colourless, even turbid, petroleum layer is determined. Of this, 50—100 c.c. are evaporated to 10—15 c.c., ether (30—40 c.c.) is immediately added, and ethyl alcohol (30—40 c.c.), the temperature being maintained at —20°. The precipitate of paraffin is rapidly separated by filtering at the pump. A solubility correction is unnecessary. The ether-alcohol mixture can be replaced by butanone. Hard and soft fractions may be determined by precipitating at 0° and then at —21°. Turbid petroleum may be neutralised with 2.5% sodium hydroxide, or mixed with fuller's earth and extracted with light petroleum.

CHEMICAL ABSTRACTS.

Origin and constitution of naphthenic acids. B. TIUTUNNIKOV (Neftjanoe Chozjajstvo, 1926, 10, 797—806).—Theories of the origin of naphthenic acids are discussed, and experimental data bearing on the problem are recorded.

CHEMICAL ABSTRACTS.

Asphalt tars. I. Terminology, methods of testing, and standards. II. Characterisation of the asphalt tars of the U.S.S.R. [Russia]. A. SACHANEN and L. SHERDEVA (Neftjanoe Chozjajstvo, 1926, 10, 393—397).

Conversion of alcohols into petroleum.—MAILKE and RENAUDIE—See III.

PATENTS.

Coke ovens. R. E. ELLIS. From FOUNDATION OVEN CORP. (B.P. 279,955, 7.8.26).—In coke ovens where the oven is tapered from the pusher to the coke side, air is admitted through a wind box at each end of the regenerator extending from the front to the back of the oven, and uniform heating is obtained by the use of ports graduated in size to the taper of the oven. The air passes through the ports to vertical combustion flues arranged in pairs or groups and connected with regenerators. Each alternate flue acts either as a combustion flue or waste-gas flue, and after combustion the waste gases pass down through graduated ports to the regenerator and on to the waste-gas flues situated at each side of the battery.

A. C. MONKHOUSE.

Generator - oven for the production of coke and gas. R. GEIPERT (G.P. 444,476, 26.10.23).—Two recuperator channels are situated at the exit from the combustion chamber; one preheats the secondary air and the other the primary air to the producer. Efficient heat economy is thereby attained.

A. B. MANNING.

Transportable apparatus for the manufacture of charcoal. A. ENDERS, R. BILLEN, and CARBONISATION INDUSTRIELLE (SOC. ANON.) (B.P. 279,709, 9.4.27).—Wood is carbonised in a retort superimposed on a furnace. The gases from the furnace pass through the retort and the combined gases after passing through condensers are led beneath the grate of a second furnace where a conversion into carbon monoxide takes place. The gases are burnt and pass up through a similar retort where they dry and subsequently carbonise the contained wood. The periods of drying and carbonisation are 2 hrs. each. Working is continuous, both furnaces act in turn as a gas generator, and the retorts are connected alternately to the condensers during the carbonisation period. When sawdust or similar material is carbonised, the retort is fitted with concentric, perforated sheet metal tubes, and the hot gases circulate in the annular spaces.

A. C. MONKHOUSE.

Utilisation of pulverulent or powdered carbonaceous materials. J. J. C. BRAND and B. LAING (B.P. 279,767, 26.5.26).—In order to ensure intimate mixing of powdered fuel and air, and to prevent a flashback from the burner, one or more gauze diaphragms are inserted in the pipe line close to the burner nozzle. The gauze can be rotated or removed for cleaning purposes.

A. C. MONKHOUSE.

Separation of solid and liquid materials [washing of coal etc.]. A. E. LEEK, and WIGAN COAL & IRON CO., LTD. (B.P. 279,525, 22.4.26 and 8.1.27).—Liquid slurry (e.g., from coal washing) is fed into a tall container capable of withstanding up to 30 lb./sq. in., and in which are a number of dished trays consisting of a perforated plate and a wire mesh. When the container is full the trays are brought into a horizontal position and the slurry kept for some time to allow the solid material to settle on the trays. Air or water pressure is supplied at the top of the container, and the slurry of each layer is filtered through a bed of material on the tray slightly coarser than the material in suspension. The clear water is withdrawn from the interior of the trays and from the bottom of the container. Fresh slurry is then pumped in and the process repeated until the trays require emptying. Double trays capable of being rotated through 180° may also be used.

A. C. MONKHOUSE.

Treatment [washing] of coal etc. WIGAN COAL & IRON CO., LTD., and A. E. LEEK (B.P. 279,766, 22.4.26).—In a coal-washing plant the slurry is removed and treated as in B.P. 279,525 (preceding) and the clean water is returned to the washing plant. Using a Wigan coal, the cake obtained from the filtered slurry contained 15% of moisture, 8% of ash, and 77% of coal.

A. C. MONKHOUSE.

Washing of coal, ores, etc. L. HOYOIS (B.P. 265,202, 26.1.27. Belg., 26.1.26. Addn. to B.P. 258,753; B., 1926, 971).—As lighter particles tend to be carried with the current of liquid through the openings through which the denser particles pass, a second trough with an adjustable partition is used and the liquid is diverted without disturbing the separation. In order to classify the products more accurately, boxes are arranged beneath the discharge orifices with auxiliary currents

of liquid which prevent the liquid in the trough and the lighter fractions passing through the orifice.

A. C. MONKHOUSE.

Apparatus for the separation of volatile products from solid carbonaceous material. R. E. COTTERMAN (U.S.P. 1,650,191, 22.11.27. Appl., 20.8.25).—Fuel is carbonised in a horizontal cylindrical retort which is heated externally. The conveyor moving the fuel along the retort is operated in conjunction with the valve regulating the feed of fuel and also the valve withdrawing the carbonised fuel. A. C. MONKHOUSE.

Apparatus for recovering the volatile products from carbonaceous material. M. J. TRUMBLE (U.S.P. 1,651,647, 6.12.27. Appl., 11.7.22).—The material is fed by conveyor to a separator to which steam is admitted, and, after removal of the volatile products, is discharged by means of a water-cooled conveyor. The preheated water is used for steam generation, and the waste heat from the separator serves to preheat the feed material.

A. C. MONKHOUSE.

Facilitating the briquetting of coal. P. PETROFF (F.P. 621,742, 22.1.26).—Powdered coal is treated with acid solutions, *e.g.*, dilute formic or acetic acid, prior to briquetting in admixture with tar, pitch, or masut.

L. A. COLES.

Pressure regulation in the hydrogenation of coal. SOC. INTERNAT. DES COMBUSTIBLES LIQUIDES (F.P. 618,674, 8.7.26. Ger., 15.12.25).—The hydrogenation is carried out at 450° under a pressure of 150–200 atm. in three chambers. The partially hydrogenated product from the first chamber passes into a separating vessel, from which the volatile products are allowed to escape; the liquid, however, passes into the second chamber in which it is further hydrogenated. The process is repeated in the third chamber. The escaping volatile products are cooled and condensed.

A. B. MANNING.

Working-up residues produced in the liquefaction of coal. SOC. INTERNAT. DES COMBUSTIBLES LIQUIDES (F.P. 618,647, 7.7.26. Ger., 11.12.25).—After removal of the light hydrocarbons and water the solid residue is treated with about 20% of an aliphatic hydrocarbon, *e.g.*, crude petroleum. After heating at 200° for 1 hr. about 40% of hard asphalt is precipitated, and after a further 3–5 hrs.' heating about 40% of a soft asphalt free from solid insoluble matter separates. A red-brown liquid oil of low asphalt content remains, and is purified by steam distillation. The same process can be applied to other bituminous material.

A. B. MANNING.

Purification of graphite by flotation. J. F. M. R. DE ROBILLARD (F.P. 621,414, 4.3.26).—The graphite is stirred with hydrocarbons and subjected to reduced pressure to remove absorbed gases under such conditions as to expose the smallest possible liquid surface, so that the graphite particles sink through a layer of water or other liquid on to a strainer.

L. A. COLES.

Production of synthetic fuel. C. LAILLET, A. CHAIX, and J. CHENAIL (F.P. 621,728, 21.1.26).—The superheated vapour of a volatile solvent such as acetone,

ether, benzene, toluene, carbon tetrachloride, petrol, etc. is passed at the ordinary pressure into a closed vessel containing powdered or granulated coal, anthracite, lignite, shale, asphalt, peat, etc., until the temperature reaches 450–500°. The solvent is recovered by evaporation from the liquid extract, and the residue is used as fuel.

L. A. COLES.

[Non-knocking] fuel composition. S. P. MARLEY and W. A. GRUSE, ASSES. to GULF REFINING CO. (U.S.P. 1,645,109, 11.10.27. Appl., 28.7.24).—An aminoaryl alkyl ether, *e.g.*, *p*-phenetidine (2.5%), is added to petrol to inhibit knocking.

T. S. WHEELER.

Fuel for internal-combustion engines. SOC. LEFRANC & CIE. (F.P. 622,077, 3.4.26).—Complex ketones prepared as described in F.P. 566,343 (*cf.* E.P. 216,120; B., 1925, 337) are added as stabilisers to mixtures of petroleum and alcohol etc.

L. A. COLES.

Working of gas retorts. KOHLENVEREDLUNG A.-G., ASSEES. of KOHLENVEREDLUNG G.M.B.H. (B.P. 267,153, 7.3.27. Ger., 5.3.26).—In the low-temperature distillation of brown coal the disadvantages due to fine dust in the fuel are overcome by removing the dust before carbonisation by means of wind sifters, sieves, or electrical dust removers. [Stat. ref. to B.P. 2513 of 1888.]

A. C. MONKHOUSE.

Gas producer. J. F. M. FAYET (F.P. 620,083, 10.8.26).—The air necessary for combustion is preheated by the hot gases and charged to a greater or less amount with water vapour before its introduction within the double walls of the producer.

A. B. MANNING.

Gas manufacture. W. S. YARD and E. N. PERCY (U.S.P. 1,649,640, 15.11.27. Appl., 4.2.24).—Carbon is heated to incandescence in a producer by means of a continuous air-blast and oil is injected on to the top of the fuel bed. The gases thus obtained then pass through particles of carbon heated by the passage of an electric current and further oil is added.

A. C. MONKHOUSE.

Recovery of gases and vapours from coke-oven gas. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G., ASSEES. of H. FRITZWEILER, W. GROB, and C. B. STUER (G.P. 444,136, 17.7.24).—The hot gases traverse a number of chambers which contain adsorbent material presenting a large surface, and which are fitted with means for their direct internal, or internal and external heating. The sensible heat of the gases is utilised in driving off the adsorbed gases and vapours from the saturated filling material in one chamber, and the gases then, before entry into the adsorbing chamber, are freed from impurities which would diminish the efficiency of adsorption.

A. B. MANNING.

Purification of gas of the kind derived from the distillation of coal or coke. "AMMONIA" (B.P. 263,830, 24.12.26. Fr., 4.1.26).—Coal gas is washed under pressure with cold water in a tower scrubber to remove carbon dioxide and hydrogen sulphide. The water leaving the scrubber is used to drive a turbine or Pelton wheel where some of the gases are evolved; the water is then pumped to a tower through which air is blown, the gases escape into the atmosphere, and the water is returned for circulation to the scrubber.

A. C. MONKHOUSE.

Gas-purification process. F. W. SPERR, JUN., and D. S. JACOBSON, Assrs. to KOPPERS Co. (U.S.P. 1,653,933, 27.12.27. Appl., 19.5.25).—Sodium thiosulphate formed in gas-purification liquors is fused in a furnace, and the product is mixed with carbonaceous material and limestone and further heated, whereby conversion into sodium carbonate takes place. H. ROYAL-DAWSON.

Apparatus for producing and treating gas. H. A. DREFFEIN (U.S.P. 1,650,614, 29.11.27. Appl., 30.9.22).—The gases are passed through a coke-filled scrubber consisting of a cylinder surmounted on and projecting into a short cylinder of larger diameter, which latter forms a circulating chamber for the gases.

A. C. MONKHOUSE.

Distillation of [carbonaceous] materials. H. B. CANNON (U.S.P. 1,651,994, 6.12.27. Appl., 11.4.24).—The finely-divided carbonaceous material falls through an elongated vertical chamber to which low-pressure saturated steam is admitted. The steam is heated by radiation from the lower walls of the chamber and effects a low-temperature distillation of the material in the upper portion.

A. C. MONKHOUSE.

Distillation of vegetable material with a high water content. M. FOURMENT (F.P. 622,052, 28.1.26).—The material is first dried and then distilled, the heat necessary for the first stage being supplied by burning the gases generated in the second stage, in which the material is heated by gas obtained from external sources.

L. A. COLES.

Suction-gas plant, producing tar as a by-product. E. MAHLKUCH (G.P. 445,334, 29.4.25).—The tar flows continuously through a still heated by the engine exhaust gases; the lighter distillation products are drawn away directly by the engine, but the heavier are separated. A separate heat supply for the still is unnecessary.

A. B. MANNING.

Preparation of lubricating oils. "REX" MINERAL-ÖLGES. STEPHAN, BOOK, & ZIEGLER (G.P. 444,958, 7.6.21).—High-boiling tar fractions are treated with alkali solutions, and, after separation of the caustic layer, are distilled, preferably *in vacuo*. The distillates are cooled to a low temperature and freed from the solid which then separates. Lubricating oils are obtained which are neutral, salt-free, stable, and miscible with mineral oils.

A. B. MANNING.

Electrically heating oils and hydrocarbons. K. HERING (G.P. 444,919, 31.1.22).—The oil passes through a coil inside an electrically-heated boiler so that the heat transmitted to the oil is dependent on the current used and on the pressure in the boiler. Overheating of the oil is thus avoided.

A. B. MANNING.

Preparation of petroleum hydrocarbons from methane. COMP. DE BÉTHUNE (F.P. 613,542, 27.7.25).—Methane or a gas containing it is heated at 200–600° under pressure with a catalyst, *e.g.*, ferric oxide, mixtures of metallic oxides, or the corresponding metals obtained by reduction of the oxides. Liquid hydrocarbons are obtained, the greater part of the product distilling between 38° and 100°. Butane and ethane are also produced. The methane used need not be pure, the presence of hydrogen, oxygen, or other gases appearing favourable to the reaction.

A. B. MANNING.

Distillation of petroleum oils. R. W. and R. J. HANNA, Assrs. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,653,431, 20.12.27. Appl., 2.10.22).—The oil is vaporised continuously and the vapours are fractionated by passage through three stages. In the first stage the vapours are subjected to the condensing action of the combined feed oil and condensate from the other stages, in the second the condensate from the third stage only is used as condensing agent, and in the third stage a separate liquid cooling medium at a controlled temperature is used. The condensates are combined with the feed oil, and the vapours are condensed as one product.

C. O. HARVEY.

Distillation of [mineral] oils and similar products. K. ZIMMERMANN (G.P. 444,985, 19.9.24).—The material is finely divided by energetic mechanical means, *e.g.*, by rotating discs, and is carried over by steam, or other gases or vapours, into a heated still filled with Raschig rings. In this way the oil can be completely vaporised without any separation of carbon. The vapours are then fractionally condensed.

A. B. MANNING.

Thermal decomposition [cracking] of hydrocarbons. G. O. CURME, JUN., Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,646,349, 18.10.27. Appl., 7.1.26).—Ferrochromium containing about 26% Cr is of value in the manufacture of cracking apparatus, since it does not catalyse the production of free carbon.

T. S. WHEELER.

Continuous process for solidifying liquid hydrocarbons. Y. DE PENIAGUA (Dutch P. 16,247, 5.1.23. Fr., 27.1.22).—Liquid hydrocarbons and a thickening agent flow continuously into a heated, well-stirred emulsion of the same materials, and a hardening agent, *e.g.*, formaldehyde, is added to the mixture in the same or in another vessel.

L. A. COLES.

Removal of sulphur from petroleum. F. C. AXTELL, Assr. to AXTELL RESEARCH LABORATORIES, INC. (U.S.P. 1,645,679, 18.10.27. Appl., 27.12.26).—Oleum is agitated with an excess of benzene, and the acid layer separating is employed to treat mineral oils containing sulphur. It has no polymerising or oxidising effects.

T. S. WHEELER.

Concentration of coal. W. W. STENNING, Assr. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,655,849, 10.1.28. Appl., 15.2.24. U.K., 16.2.23).—See B.P., 215,841; B., 1924, 587.

Production of stable aqueous emulsions of pitch and other saponified organic matter of mineral origin. F. C. THORNLEY, F. F. TAPPING, and O. REYNARD, Assrs. to THORNLEY & Co. (U.S.P. 1,653,026, 20.12.27. Appl., 21.7.23. U.K., 26.1.23).—See B.P. 219,348; B., 1924, 780.

Manufacture of mixed gas. H. NIELSEN and J. R. GARROW (U.S.P. 1,654,942, 3.1.28. Appl., 21.3.21. U.K., 23.2.20).—See B.P. 162,459; B., 1921, 425 A.

Treatment of fuel gas. E. H. BIRD, Assr. to KOPPERS Co. (U.S.P. 1,654,782, 3.1.28. Appl., 2.1.23).—See B.P. 209,379; B., 1924, 588.

[Paraffin] wax sweating and crystallising apparatus. H. L. ALLAN and J. MOORE, Assrs. to BURMAH

OIL CO., LTD. (U.S.P. 1,654,232, 27.12.27. Appl., 9.12.24. U.K., 29.8.24).—See B.P. 243,447; B., 1926, 147.

Removal of tar acids from ammonia liquor and other liquors. H. W. ROBINSON and D. W. PARKES (U.S.P. 1,653,783, 27.12.27. Appl., 23.6.26. U.K., 7.8.25).—See B.P. 260,686; B., 1927, 39.

Oil still. J. PRIMROSE, ASST. to POWER SPECIALTY CO. (U.S.P. 1,654,347, 27.12.27. Appl., 28.12.20).—See B.P. 179,493; B., 1922, 489 A.

Apparatus for generating acetylene under pressure. R. MÜTSCHLE (B.P. 271,803, 24.7.26. Ger., 25.5.26).

Conducting catalytic reactions (G.P. 445,252).—See I. **Purification of crude naphthalene** (F.P. 583,270).—See III. **Ammonium chloride etc.** (G.P. 426,389). **Ammonium sulphate crystals** (B.P. 255,876). **Lead tetraethyl** (U.S.P. 1,645,375). **Lead tetra-alkyl** (U.S.P. 1,645,389 and 1,645,390). **Hydrogenation of hydrocarbons** (F.P. 621,902).—See VII. **Pitch compositions** (B.P. 256,640).—See IX.

III.—ORGANIC INTERMEDIATES.

Conversion of alcohols into petroleum spirit. A. MAILHE and RENAUDIE (Compt. rend., 1927, 185, 1598—1600).—When *n*-butyl alcohol vapour is passed over uranium oxide at 420—440° it yields gaseous products consisting of carbon dioxide, 11%; carbon monoxide, 5.8%; hydrocarbons C_nH_{2n} , 16.8%; hydrocarbons C_nH_{2n+2} , 30.6%; and hydrogen, 35.8% when a fresh catalyst is used (the corresponding proportions being 4.9%, 9.5%, 28.5%, 35.3%, and 21.3% respectively, with a used catalyst) and volatile liquid products consisting of butaldehyde and mixtures of paraffin and olefine hydrocarbons of b.p. ranges from 80° to 125°. These contain six or more atoms of carbon, and are probably formed by a crotonaldehyde type of condensation of the butaldehyde followed by loss of the carbonyl residue and fission of the hydrocarbons $CHPr:CHEt$ and $CHPr:CEt:CH:CHEt$ so obtained into lower homologues, which may be partially reduced to the corresponding saturated hydrocarbons by the hydrogen which is present. J. W. BAKER.

PATENTS

Production of formaldehyde. W. C. ARSEM, ASST. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,648,602, 8.11.27. Appl., 4.8.26).—Formic acid vapour mixed with carbon dioxide is passed at 300—500° and 10—100 atm. pressure over a basic chloride or sulphate of zinc, tin, or magnesium. T. S. WHEELER.

Concentrating aqueous solutions of acetic or formic acid. W. N. HAWORTH and J. NELSON, LTD. (B.P. 281,827, 19.10.26).—Aqueous acetic acid mixed with anhydrous sodium (or potassium) acetate, or formic acid mixed with anhydrous sodium formate, is distilled under reduced pressure. In each case the bulk of the water distils off, and then on raising the temperature strong acid passes over. In examples, over 80% of 30% acetic acid is recovered as 98.6% acid, and over 80% of 30% formic acid as 98% acid. The operation is conducted in a single still provided with means to break the lumps of solid. B. FULLMAN.

Preparation of halogeno-fatty acid anhydrides. H. T. CLARKE and C. J. MALM, ASSTS. to EASTMAN KODAK CO. (U.S.P. 1,648,540, 8.11.27. Appl., 10.12.26).—A halogeno-fatty acid (*e.g.*, chloroacetic acid) (2 mols.) is heated with acetic anhydride (1 mol.), the acetic acid produced being distilled off as formed.

T. S. WHEELER.

Manufacture of unsymmetrically substituted diaminopropanols [β -hydroxypropylenediamines]. I. G. FARBERIND. A.-G. (B.P. 275,622, 3.8.27. Ger., 3.8.26).—Compounds of the type, $NH_2 \cdot CH_2 \cdot CH(OH) \cdot CH_2 \cdot NRR'$, are obtained from epichlorohydrin by the successive action of a secondary amine and ammonia. From diethylamine and epichlorohydrin there is obtained (*diethylaminomethyl*)ethylene oxide, b.p. 155—159°, or 40—50°/8 mm., which reacts with ammonia to give β -amino- β' -diethylaminoisopropyl alcohol, b.p. 223°, or 120—130°/30 mm., and with ethylamine to give triethyl- $\beta\beta'$ -diaminoisopropyl alcohol, b.p. 230—232°, or 100—120°/6 mm. (*Piperidinomethyl*)ethylene oxide, b.p. 72—77°/8 mm., β -amino- β' -piperidinoisopropyl alcohol, b.p. 148—150°/29 mm., (*methylanilinoethyl*)ethylene oxide, b.p. 132—135°/8 mm. or 160—162°/30 mm., and β -amino- β -methylanilinoisopropyl alcohol [*methyl- γ -amino- β -hydroxypropylaniline*], m.p. 71°, b.p. 205—210°, are similarly prepared. The diamino-alcohols are intermediates for therapeutic substances. C. HOLLINS.

Preparation of substituted guanidines. E. KLINE, ASST. to GRASELLI CHEMICAL CO. (U.S.P. 1,648,184, 8.11.27. Appl., 6.10.24).—In the preparation of substituted guanidines, *e.g.*, diphenylguanidine, from the corresponding thiocarbamides by the action of a desulphurising agent in presence of alcoholic ammonia, the use of a water-soluble lead salt, *e.g.*, lead nitrate, as the desulphurising agent, is claimed. T. S. WHEELER.

Production of benzoic acid from phthalic anhydride. C. CONOVER, ASST. to MONSANTO CHEMICAL WORKS (U.S.P. 1,645,180, 11.10.27. Appl., 21.12.25).—Phthalic anhydride vapour and steam (50 pts.) are passed at 200—600° over zinc, copper, or aluminium oxide, to form benzoic acid in 90% yield. T. S. WHEELER.

Production of adipic acid and its alkyl substitution products. J. D. RIEDEL A.-G. (B.P. 265,959, 7.2.27. Ger., 12.2.26).—Catalysts (ammonium vanadate, mercuric oxide, molybdenum pentoxide) enable the oxidation of cyclohexanols or cyclohexanones to adipic acids by nitric acid to take place at 50—65° with production of fewer by-products. C. HOLLINS.

Purification of hydrocarbon compounds by hydrogenation and reduction. SOC. INTERNAT. DES COMBUSTIBLES LIQUIDES (F.P. 618,490, 5.7.26. Ger., 2.12.25).—Unstable hydrogenated organic compounds, or those readily hydrogenated, are used as hydrogen carriers. Inorganic compounds, *e.g.*, alkaline ferric oxide, or "lux," which catalyse the reaction may also be added. The readily oxidisable organic hydrogenated compounds are prepared as follows. Anthracene and solid potassium hydroxide are heated with hydrogen under pressure. At about 400° an active product is formed as a crystalline paste, which, on being distilled until the appearance of yellow fumes, yields 80—85% of a hydrogenated anthracene. This product acts as a

hydrogen carrier when heated with a "flaming" coal under 100 atm. pressure at 390° for 10 min. The pressure falls and on cooling to 15° reaches a final value of 65 atm. The yield is about the same as after 1 hr.'s heating, but the product with the shorter reaction time is of better quality and possesses a lower density and a smaller phenol content.

A. B. MANNING.

Conversion of phenols into hydrocarbons. SOC. INTERNAT. DES COMBUSTIBLES LIQUIDES (F.P. 618,520, 6.7.26. Ger., 2.12.25).—The phenol or mixture of phenols is heated at 470° with hydrogen under 90 atm. pressure for 1 hr. To obtain the maximum reduction the light oils and water formed should be removed continuously from the autoclave. The process yields 80% of light hydrocarbons distilling to 250°. Methane and water are formed as by-products. The unchanged phenol can be again submitted to the process.

A. B. MANNING.

Reduction of nitro-compounds [to hydrazo compounds]. R. A. NELSON and A. PRASIL, Assrs. to NAT. ANILINE & CHEMICAL Co., INC. (U.S.P. 1,644,483—4, 4.10.27. Appl., 22.6.20).—(A) An aromatic nitro-compound, e.g., *o*-nitroanisole, in alcoholic solution is treated gradually with zinc dust and a limited amount of sodium hydroxide solution. The product is recovered by distilling off alcohol and extracting the residue with benzene. (B) The nitro-compound dissolved in benzene is treated as in (A).

T. S. WHEELER.

Purification of crude naphthalene. OBERSCHLESISCHE KOKSWERKE & CHEM. FABR. A.-G., F. RUSSIG, and P. DAMM (F.P. 583,270, 3.7.24).—The impurities are polymerised by treatment with a suitable reagent, e.g., sulphuric acid, ferrous chloride, etc., their b.p. being thereby so far raised that the pure naphthalene can be separated by distillation or sublimation. E.g., the hot-pressed crude product is shaken for ½ hr. with 0.5% of sulphuric acid, the latter then neutralised, and the pure naphthalene recovered by vacuum distillation or sublimation. If ferrous chloride is used the mixture can be sublimed directly.

A. B. MANNING.

Manufacture of 2-aminoanthraquinone. D. G. ROGERS, Assr. to NAT. ANILINE & CHEMICAL Co., INC. (U.S.P. 1,644,494, 4.10.27. Appl., 9.11.22).—The production of aminoanthraquinones from the corresponding sulphonic acids by the action of ammonia solution in presence of an oxidising agent, e.g., nitrobenzene, is facilitated by addition of an ammonium salt, e.g., ammonium chloride.

T. S. WHEELER.

Manufacture of new derivatives of 2:3-hydroxynaphthoic arylamides. BRIT. DYESTUFFS CORP., LTD., J. B. PAYMAN, and H. WIGNALL (B.P. 281,795, 20.9.26).—2:3-Hydroxynaphthoic acid is condensed in presence of phosphorus trichloride etc. with an aminoarylsulphonamide to give hydroxynaphthamidoarylsulphonamides of the type, $\text{OH}\cdot\text{C}_{10}\text{H}_6\cdot\text{CO}\cdot\text{NR}\cdot\text{Ar}\cdot\text{SO}_2\cdot\text{NR}''$, where R, R', and R'' may be hydrogen or hydrocarbon radicals. *m*-2-Hydroxy-3-naphthamidobenzenesulphonamide ($\text{R} = \text{R}' = \text{R}'' = \text{H}$), m.p. 278°, is made from 2:3-hydroxynaphthoic acid, aniline-*m*-sulphonamide, and phosphorus trichloride in toluene at 60°. The preparation of *p*-toluidine-3-sulphonanilide, methylaniline-*p*-sulphonamide, and ethyl-*o*-toluidine-4-sulphonanilide is mentioned; these

and aniline-*m*-sulphonanilide are also condensed with 2:3-hydroxynaphthoic acid. The isolated hydroxynaphthoyl chloride may be used.

C. HOLLINS.

Manufacture of 2:3-hydroxynaphthoic acid. W. S. CALCOTT, A. R. HITCH, and H. W. MAHR, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,648,839, 8.11.27. Appl., 23.2.24).—Sodium β -naphthoxide is treated at 225–235° with carbon dioxide at 30–40 atm. pressure.

T. S. WHEELER.

Manufacture of 1-aminonaphthalene-8-carboxylic acid [8-amino- α -naphthoic acid]. R. HERZ and F. SCHULTE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,646,290, 18.10.27. Appl., 12.7.26. Ger., 29.11.24).—See E.P. 276,126; B., 1927, 808.

Production of new *N*-alkylcarbazolephosphinous acids. A. VON WEINBERG and W. SCHMIDT, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,648,207, 8.11.27. Appl., 1.10.25. Ger., 7.10.24).—See B.P. 258,744; B., 1926, 996.

Oxidation of aldoses (U.S.P. 1,648,368)—See XVII.

IV.—DYESTUFFS.

Cacao-red. A. HEIDUSCHKA and B. BIENERT (J. pr. Chem., 1927, [ii], 117, 262–272).—Complete analyses of the cacao-bean, kernel, shell, and embryo have been made. Complete details for the extraction of cacao-red (cf. Schweitzer, A., 1899, i, 300; Reutter, *ibid.*, 1913, i, 1031; 1914, ii, 760) are given, together with the solubility of this substance in numerous organic solvents. When heated with water or dilute sulphuric acid, at 130–140°, there is formed a sugar (cf. Reutter, *loc. cit.*) which reduces Fehling's solution, gives a positive reaction for aldehyde with diazobenzenesulphonic acid, a violet coloration with α -naphthol and sulphuric acid, etc., and also a brown product resembling humic acid. Hydrolysis with potassium hydroxide solution yields acetic and protocatechuic acids, and probably dimethylphloroglucinol, m.p. 165° after browning at 150–160°. The colouring matter can be acetylated or benzoylated, and yields, on distillation with zinc dust, an unidentifiable oil. The formula $(\text{C}_{17}\text{H}_{16}\text{O}_7)_n$ is deduced, and the suggestion made that the compound is a polymeric 3:5:7:3':4'-pentahydroxy-6:8-dimethyl-2:3-dihydroflavonol. The brown compound obtained by the action of dilute sulphuric acid is probably a dehydrated flavonol.

H. BURTON.

Microchemical detection of Orange II. L. SOEP (Chem. Weekblad, 1927, 24, 623).—Several microchemical reactions of Orange II have been examined. The thallium salt forms in red needles, grouped starwise, which appear blood-red in polarised light.

S. I. LEVY.

Alizarin lakes. WEISER and PORTER. See XIII.

PATENTS.

New secondary disazo dyes [for viscose silks]. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, P. CHORLEY, and R. BRIGHTMAN (B.P. 281,767, 8.9.26).—Aminoazo compounds are diazotised and coupled in acid or alkaline media with sulphonic acids of 2:8-aminonaphthols, $\text{OH}\cdot\text{C}_{10}\text{H}_6\cdot\text{NHR}$, in which R is an aryl group of the benzene series or a carboxylic acyl group, but contains no amino-substituent. Black, brown, blue, and grey

level shades are obtained on viscose silks. Examples are: Aniline \rightarrow Cleve acid \rightarrow 4-*m*-xylyl- γ -acid [2-(4-*m*-xylylamino)-8-naphthol-6-sulphonic acid]; metanilic acid \rightarrow α -naphthylamine \rightarrow phenyl- γ -acid; technical aminosalicic acid \rightarrow α -naphthylamine \rightarrow benzoyl- γ -acid; *p*-aminoazobenzenesulphonic acid \rightarrow phenyl- γ -acid. Ten other couplings are listed. C. HOLLINS.

Preparation of 2-hydroxybenzanthrone. I. G. FARBERIND. A.-G., Assecs. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (B.P. 258,910, 28.9.26. Ger., 28.9.25. Addn. to B.P. 224,522; B., 1925, 583).—The nitro-group in 2-nitrobenzanthrone-3-diazonium sulphate is rapidly replaced by hydroxyl under the conditions of diazotisation, and the diazo group may be eliminated by means of boiling alcohol without special formation of the diazo-oxide. C. HOLLINS.

Manufacture of halogen derivatives of aromatic compounds. J. H. CROWELL, Assr. to NAT. ANILINE & CHEMICAL Co., Inc. (U.S.P. 1,646,235, 18.10.27. Appl., 26.9.24).—Anthraquinone vat dyes can be readily halogenated by treatment with chlorine or bromine in presence of sulphur dioxide and a suitable organic solvent or diluent, *e.g.*, nitrobenzene. T. S. WHEELER.

Method of effecting caustic fusions [to produce indigo]. D. G. ROGERS, Assrs. to NAT. ANILINE & CHEMICAL Co., Inc. (U.S.P. 1,644,493, 4.10.27. Appl., 17.6.21).—The caustic fusion of substances which yield indigo, *e.g.*, phenylglycine, is performed with a reduced amount of alkali in presence of a petroleum hydrocarbon liquid at ordinary temperatures and boiling above 200°. T. S. WHEELER.

Manufacture of benzanthrone derivatives containing sulphur. H. NERESHEIMER and H. EMMER, Assrs. to I. G. FARBERIND. A.-G. (U.S.P. 1,644,851, 11.10.27. Appl., 9.2.26. Ger., 7.3.25).—See B.P. 255,731; B., 1926, 867.

Preparation of 2-(or 3-)chloroquinizarin. I. GUBELMANN, Assr. to NEWPORT Co. (U.S.P. 1,655,863, 10.1.28. Appl., 2.11.25).—See B.P. 260,544; B., 1927, 838.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Cooking of spruce chips with sulphur dioxide solutions of high concentration. J. PALMEN (Pulp and Paper Mag., 1927, 25, 1547—1551).—A series of experimental cooks was made to determine the effect of high concentrations of sulphur dioxide (obtained by the use of ethyl alcohol as solvent) on the resolution of spruce wood. Acid liquors containing from 70.8% of alcohol and 26.5% of sulphur dioxide to 11.5% of alcohol and 4.6% of sulphur dioxide with addition of 0.7% of ammonia or up to 3.2% of lime were used at maximum temperatures varying from 105—136°. The results of these experiments indicate that (a) a black discoloration is characteristic of pulps cooked in the presence of alcohol, though the colour is removed to a large extent on washing with water, and in some cases an absolutely white pulp results; (b) the use of liquors containing high percentages of sulphur dioxide does not (in the presence of alcohol) effect any considerable reduction of the cooking time or temperature; and (c) a high percentage of alcohol slows down the cooking action

very considerably, the pulp obtained with a cooking liquor containing 70.8% of alcohol showing a lignin content of 23.8% after an effective cooking time of 18 hrs. with a maximum temperature of 130°.

D. J. NORMAN.

Caroá fibre as a papermaking material. M. B. SHAW and G. W. BICKING (U.S. Bur. Stand. Tech. Paper No. 340, 1927, 21, 323—346).—Experimental, followed by semi-commercial, cooks indicate that caroá fibre could be satisfactorily used for papermaking. The yield, soda consumption, and the character of the pulp depend to some extent on the preliminary treatment of the raw material. Retting appears to give the most favourable results, a 55.3% yield of pulp (dry pulp on dry material after cutting and dusting) being obtained by digestion with 25% of caustic soda for 4 hrs. at 172°. When bleached with 10% of bleaching powder the product compares favourably with rag pulp and is capable of furnishing paper of high strength. The semi-commercial trials were made on unretted fibre that had been beaten dry, and gave lower yields of bleaching pulp, *e.g.*, 49.5—51.5%. Caroá fibre has a fibre length of 2.25—5.75 mm. (average 4 mm.) and a thickness of 0.0046—0.0154 mm. (average 0.01 mm.), and should therefore have good felting properties. When less soda is used pulps are obtained which, though technically unbleachable, provide excellent wrapping papers equal in strength to those made from kraft pulp. There is no organised caroá industry at present, but the economic availability of the fibre for papermaking appears promising. The papermaking equipment and the technique employed in papermaking research are described in detail.

D. J. NORMAN.

Characterisation of cellulose preparations by the rotation method. E. HÄGGLUND and F. W. KLINGSTEDT (Annalen, 1927, 459, 26—38).—The optical rotation curves in cuprammonium solution for various specimens of cellulose have been plotted. The curves obtained in accordance with the method of Hess, Messmer, and Ljubitsch (A., 1925, i, 1246) with (a) cotton containing 95.2% of α -cellulose, ash content 0.19%, and free from pentosans and mannosans, (b) wood cellulose containing 87.7% of α -cellulose, 0.3% of ash, 1.9% of xylosan, and 3.2% of mannosan in the ash-free sample, and (c) mercerised cellulose which, owing to its treatment with alkali, is free from pentosans and mannosans, are almost identical, and the mean percentage deviations from the curve obtained with pure cellulose prepared from the crystalline acetate by the method of Hess are only 7.68%, 5.16%, and 7.49%, respectively. Xylosan itself in cuprammonium solution shows a higher levorotation than cotton cellulose. The authors, therefore, do not agree that this method is a reliable one for establishing the identity of cellulose preparations from different sources, or for following the course of the purification of the specimens.

J. W. BAKER.

Two natural processes of decomposition of the cellulose and lignin of lignified tissue by bacteria. R. FALCK (Cellulosechem., 1928, 9, 1—6; cf. Falck and Haag, B., 1927, 213).—In the decomposition of grain straw by the bacteria present in horse dung two distinct processes are observed. With a large mass of material bacteriological "destruction" takes place with evolu-

tion of heat, but if the temperature be lowered by laying out the material in small beds, a different type of decomposition—bacteriological “corrosion”—sets in. The effect of “destruction” on straw is to reduce the cellulose content and, more rapidly, the pentosan content, whilst the lignin content increases owing to the removal of cellulose, since the lignin is practically unaffected by this process. The amount of moisture present decreases, and the carbon content rises chiefly on account of the removal of cellulose, the residue being mainly lignin. The amount of alkali-soluble material present decreases and considerable gas evolution occurs. Carbon dioxide together with some ammonia is evolved, but methane is not formed in appreciable quantities even in the centre of the heap. Bacteriological corrosion, as in the case of wood corrosion (Falck and Haag, *loc. cit.*), results in the slow decomposition of both lignin and cellulose, and the pentosan content falls off at the same rate as that of cellulose. The carbon content decreases, carbon dioxide is continuously evolved, and nitrates are formed in appreciable quantity.

W. J. POWELL.

Economy of the sulphite-cellulose cooking process. A. FROBERG (Papier-Fabr., 1928, 26, 17—22).—Possibilities for more economical working in the German cellulose industry are surveyed since progress lies chiefly in increased production and the cheapening of the final product. All cookers should be fitted with mechanical filling devices, and the processes of filling, steaming, lye treatment, and emptying should be shortened as much as possible. No great economy results from steam or sulphur recovery, but increase in the capacity of the cooker is advantageous. Improvement is best made in connexion with the wood, which accounts for 50% of the total costs. Higher cooking velocity can be obtained by increasing the concentration of acid and the temperature, but yields are poorer and the quality of the cellulose is affected. Further investigation into the mechanism of the cooking process is necessary. Kinetically, the latter depends on concentration and temperature (although the diffusion of sulphur dioxide and salts into the wood must also be considered), and it may be conceived as the hydrolysis of hemicellulose, together with the partial hydrolysis and saponification of lignin. It is uncertain at what stage sulphonation of the lignin occurs, but the latter reaction is slower than the hydrolysis. The velocity of decomposition of the wood is determined by the free hydrogen ions, which are proportional to the concentration, whilst the dissociation of the acid is influenced by the lime content. The decomposition of wood by sulphurous acid is similar to the inversion of sucrose and the hydrolysis of starch, and just as at high temperatures organic acids (such as formic and acetic) are produced with decomposition of the sugar, so with wood cooked at above 140° organic acids are formed, a smaller yield of cellulose is obtained, and its strength is reduced.

B. P. RIDGE.

Economic production of strong mechanical [and chemical] wood pulp of good colour. L. ENGE (Papier-Fabr., 1928, 26, 1—3).—Ground wood, which becomes coloured when heated with steam, yields a pulp of good colour by this process when a small proportion of hydrochloric or sulphuric acid (1 in 3000—

10,000 pts. of steam) is introduced and the pulp is cooled and washed in the digester with the least possible exposure to air, the colour being further improved by the addition of a small percentage of acid to the wash water. Good results are also obtained by treatment with sulphur dioxide in the digester, through which steam is then circulated, a concentrated solution of sodium sulphite being introduced by means of a spray. A wood pulp substitute may be prepared from wood in the form of chips by heating at 100° for 6—8 hrs. with sulphur dioxide and steam, and the product, which is readily pulped and remains white, is still further improved by the introduction of a small quantity of hydrogen chloride into the gaseous mixture. Sawdust, after treatment first with sulphur dioxide and then with steam and concentrated sodium hydroxide solution introduced through a spray (15 kg./m.³ of digester), yields a completely pulped product.

W. J. POWELL.

Determination of the copper number of paper. B. W. SCRIBNER and W. R. BRODE (U.S. Bur. Stand. Tech. Paper No. 354, 1927, 22, 9—14).—The method is a modification of those of Gault and Mukerji (B., 1924, 289) and of Staud and Gray (B., 1925, 841). Fine subdivision of the sample to pass 7-mesh is desirable in all cases and is essential with heavy, gelatin-sized papers if uniform and accurate results are to be obtained. A suitable grinder which has the advantage of not heating the paper is that described by Köhler and Hall (B., 1926, 268). 25 c.c. of copper sulphate solution (277.3 g. of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ dissolved in 4 litres of distilled water), 25 c.c. of tartrate solution (150 g. of sodium hydroxide and 500 g. of sodium potassium tartrate dissolved in 1500 c.c. of distilled water), and 125 c.c. of water are heated at 100° in a 30% calcium chloride bath maintained at 115—120°. 1.5 g. of the finely-ground sample are introduced and the mixture is gently boiled, with stirring, for about 30 min. The solution is then filtered hot through paper, and the residue, after washing with 250 c.c. of hot water, is treated with 25 c.c. of molybdate solution (100 g. of sodium molybdate [43% Mo], 75 c.c. of phosphoric acid [83%], 275 c.c. of concentrated sulphuric acid, and 1750 c.c. of distilled water). After keeping for a short time the mixture is diluted with 25 c.c. of water and stirred to disintegrate the filter paper, which is collected on a filter and washed with 200 c.c. of cold water. The filtrate and washings are finally titrated with standard permanganate solution (1.5625 g. per litre).

D. J. NORMAN.

[Caustic] soda recovery plant [for use with waste-liquors from esparto grass digestion]. J. HOLMES (Proc. Tech. Sect. Papermakers' Assoc., 1926, 7, 27—45).—The low heat efficiency of soda recovery units of the usual design is discussed, and a description is given of the Holmes closed system of recovery in which a considerable economy of heat is realised by blowing the digester charge into a closed pressure tank, which also receives the relief steam from the digester during the cook. Feed liquor to the evaporator is withdrawn from the bottom of this tank, and the steam available from the pressure reduction is used to heat the first effect of the evaporator. Heat exchangers are placed in the vapour pipes of the evaporator to effect

the transfer of heat to the wash liquor and to fresh caustic liquor. The operation of the vacuum evaporator is dealt with in detail, and emphasis is laid on the reduction of efficiency caused by air leaks, gases dissolved in the liquor, and scale. The prospect of utilising the heat, which under the present system is dissipated in the roaster, is also discussed, and it is suggested that, given reasonably efficient plant, the total heat recovered should supply not only the soda recovery plant, but also the digesters.

D. J. NORMAN.

PATENTS.

Utilisation of bark and wood waste for the production of cellulose. C. ALLÈGRE, H. BRUNEL, G. P. GALINOU, and J. E. LAURIAC (F.P. 622,007, 20.1.26).—The material, after it has been softened in water, is treated with chlorine for $1\frac{1}{2}$ hrs. in a closed vessel, and is then washed successively with water, sodium sulphite solution, and again with water. The process is repeated as often as is necessary.

L. A. COLES.

Manufacture of cellulose esters of organic acids. C. J. MALM, Assr. to EASTMAN KODAK Co. (U.S.P. 1,645,915, 18.10.27. Appl., 23.9.26).—The acylation, e.g., acetylation, of cellulose is catalysed by perchlorates, e.g., of copper, zinc, or magnesium.

T. S. WHEELER.

Utilisation of [recovery of plasticising and softening agents from] cellulose acetate waste. P. D. ARON (F.P. 622,074, 30.1.26).—The material is treated with solvents, e.g., trichloroethylene, alcohol, etc., which dissolve the plasticising and softening agents, but not cellulose acetate.

L. A. COLES.

Changing [lowering] the viscosity characteristics of nitrocellulosic material. P. C. SEEL, Assr. to EASTMAN KODAK Co. (U.S.P. 1,648,509, 8.11.27. Appl., 11.2.25).—Nitrocellulose is immersed in aqueous methyl-alcoholic pyridine solution at room temperature for 24 hrs.

T. S. WHEELER.

Production of viscose. F. KEMPTER (U.S.P. 1,656,120, 10.1.28. Appl., 25.8.24. Ger., 14.5.24).—See B.P. 234,039; B., 1925, 914.

Manufacture and spinning of derivatives of cellulose and formic acid. J. G. JURLING, Assr. to FABR. VAN CHEM. PRODUCTEN (U.S.P. 1,656,199, 10.1.28. Appl., 29.5.26. Holl., 4.6.25).—See B.P. 260,650; B., 1927, 103.

Precipitation of artificial threads, ribbons, films, etc. of viscose. A. KÄMPF (U.S.P. 1,654,818, 3.1.28. Appl., 27.1.26. Ger., 6.8.21).—See B.P. 184,450; B., 1923, 711 A.

Manufacture of chemical wood pulp. A. PEETZ (U.S.P. 1,653,730, 27.12.27. Appl., 29.1.25. Ger., 30.4.24).—See B.P. 233,318; B., 1925, 800.

Purification of zinc solutions (B.P. 263,809).—See VII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

[Printing of] reserves under indanthrene dyes. R. HALLER (Sealed Note No. 1768, 4.10.07. Bull. Soc. Ind. Mulhouse, 1927, 93, 497—498). Report by P. WERNER (*Ibid.*, 498—501).—Reserves under indanthrene dyes obtained by means of copper sulphate are improved

by the addition of picric acid to the reserve paste. *E.g.*, oil-prepared cotton fabric is printed with a reserve paste consisting of 1 litre of a 50% aqueous solution of gum, 200 g. of copper sulphate, 400 g. of water, 200 g. of picric acid, and 200 g. of pipe clay (talc powder), then stored for 1—2 days, slop-padded at 65° with a dye liquor consisting of 30 litres of water, 900 g. of Indanthrene (Blue) S double (B.A.S.F.), 15 g. of Flavanthrene (Yellow) R, 2.25 litres of caustic soda (*d* 1.261), and 4.5 litres of a solution of sodium hyposulphite (660 g. of sodium hyposulphite, 5 litres of water, and 350 g. of caustic soda, *d* 1.261), washed immediately, passed through dilute sulphuric acid (*d* 1.075), soaped, and dried. Coloured reserves may be obtained by means of direct dyes using a reserve paste consisting of 0.25 litre of Madras gum solution, 50 g. of picric acid, 50 g. of talc, 70 g. of water, and 30 g. of a direct dye (Chloramine Yellow, or Chloramine Orange, or Azophor Rose A). Werner reports favourably on the process, but points out that more satisfactory processes have been devised subsequently.

A. J. HALL.

Dyeing of dog skins. MACH.—See XV.

PATENTS.

Dyeing [regenerated cellulose silks] with azo dyes. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, P. CHORLEY, and R. BRIGHTMAN (B.P. 281,410, 7.9.26).—Viscose silks are dyed in level brown, blue, red, or violet shades by means of azo dyes having as end-components 2:8-aminonaphtholsulphonic acids, particularly 2-amino-8-naphthol-6-sulphonic acid ("γ-acid"), and their *N*-derivatives. Examples are: *p*-Chloro-aniline → γ-acid (reddish-brown); 5-nitro-*o*-anisidine → phenyl-γ-acid (blue); aniline → α-naphthyl-γ-acid (brown); *p*-nitroaniline-*o*-sulphonic acid → phenyl-γ-acid ← α-naphthylamine (blue-violet), etc. The preparation of 8-hydroxy-2:2'-dinaphthylamine-6-sulphonic and -3:6-disulphonic acids by the bisulphite method is described.

C. HOLLINS.

Dyeing and printing cellulose esters and ethers. I. G. FARBERIND. A.-G. (B.P. 269,934, 23.4.27. Ger., 23.4.26).—Non-phototropic yellow dyeings on acetate silk etc. are obtained by using monoazo dyes derived from a *p*-substituted phenol as coupling component, and containing not more than one sulphonic group. Examples are: *p*-phenylenediamine, *p*-aminoacetanilide, *p*-phenylenediaminesulphonic acid, or *m*-chloroaniline, coupled with 1 mol. of *p*-cresol.

C. HOLLINS.

Dyeing of cellulose esters. SOC. CHEM. IND. IN BASLE (GES. F. CHEM. IND. IN BASEL) (B.P. 261,423, 15.11.26. Switz., 14.11.25).—Fast blue-green to green shades on acetate silk are obtained by using indophenols in which the oxygenated nucleus carries one or more halogen atoms. Examples are the indophenols made by oxidation of 2-chloro-, 2:6-dichloro-, and 2:5:6-trichloro-*p*-aminophenols with dimethylaniline, diethylaniline, benzylmethylaniline, *N*-phenyl-*N*-ethylglycine, methyl-diphenylamine, *o*-toluidine, α-naphthylamine, etc. The di- and tri-chloro- are greener than the monochloro-compounds.

C. HOLLINS.

[Mercerisation etc. of] natural vegetable fibre. BRIT. DYESTUFFS CORP., LTD., and A. J. HAILWOOD (B.P. 281,473, 30.11.26).—In the mercerisation, parch-

mentisation, or linnenisation of cotton, all or part of the sodium hydroxide used may be replaced by a sulphonium hydroxide, *e.g.*, trimethylsulphonium hydroxide.

C. HOLLINS.

Dyeing of artificial silk. J. BADDILEY, P. CHORLEY, and C. BUTLER, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,653,757, 27.12.27. Appl., 2.4.27. U.K., 7.6.26).—See B.P. 276,757; B., 1927, 841.

Treatment of textile fibres, yarns, fabrics, etc. for obtaining effects of colour or lustre or both. H. J. J. JANSSEN, Assr. to N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (U.S.P. 1,653,962, 27.12.27. Appl., 28.5.25. Holl., 7.4.25).—See F.P. 597,231; B., 1926, 437.

Fast dyeing on the fibre. T. KIRCHEISEN, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,646,296, 18.10.27. Appl., 12.10.25. Ger., 27.9.24).—See B.P. 262,537; B., 1927, 104.

Reserve dyeing on vegetable fibres. H. LEEMANN and G. TAGLIANI, Assrs. to MUNITEX CORP. (U.S.P. 1,649,710, 15.11.27. Appl., 27.4.25. Ger., 7.5.24).—See B.P. 233,704; B., 1925, 956.

Steaming or ageing machines for dyeing and like operations. J. S. WILSON, G. W. SHEARER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 282,133, 9.9.26).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Denitration of waste acids under diminished pressure. A. SCHMID (Z. ges. Schiess- u. Sprengstoffw., 1927, 22, 354—358).—By the external heating of a waste acid under diminished pressure it is possible to avoid the dilution of the recovered sulphuric acid by water condensed from the live steam usually employed as a heating agent. Under suitable working conditions a water-white sulphuric acid, free from nitrogen oxides, of a 15% higher concentration can be obtained. The apparatus consists of a horizontal thermisilid retort at one end of which is a packed dephlegmator through which the waste acid is introduced. The part of the retort near this dephlegmator is heated, electrically if possible, otherwise by oil rather than by coal or coke. The acid passes along the retort to the end away from the dephlegmator and then returns through tubes arranged in the retort which act as a heat exchanger. The nitric acid from the waste acid passes through the dephlegmator and is condensed in a coil condenser. The nitrous gases and excess air pass to a tower system where the nitrous gases are recovered as dilute nitric acid. Owing to the smaller amount of reduction during denitration, the towers are smaller than those used in the ordinary denitration system.

S. BINNING.

Substitution of sodium for potassium compounds [as reagents]. A. R. SMITH and F. C. VILBRANDT (J. Elisha Mitchell Sci. Soc., 1926, 42, 118—121).—Sodium, potassium, barium, and calcium permanganates give identical results. Sodium iodide, hydroxide, chromate, dichromate, cyanide, sulphide, sulphate, nitrate, chlorate, and ferrocyanide may be substituted for potassium compounds in appropriate analytical methods. Alcoholic sodium hydroxide may replace potassium hydroxide

in determining the saponif. values of fats and oils, and for determining methyl esters in menthol, but not for determining the saponif. values of beeswax and rosin.

CHEMICAL ABSTRACTS.

Hydrogen in gas mixtures. BEET.—See II.

PATENTS.

Manufacture of sulphuric acid. H. PETERSEN (B.P. 281,551, 7.6.27).—Acid manufactured in towers etc. filled with small grains of quartzite is denitrated in one of the towers by sulphurous gases from the roasting ovens after they have been used for acid concentration, and are therefore partially cooled. The gases may be further cooled in a cooling and condensation apparatus to utilise their heat still more and to eliminate moisture.

W. G. CAREY.

[Reaction enclosures for use in the] manufacture of sulphuric acid. A. SHARP. From Soc. GÉN. MÉTALLURGIQUE DE HOBOKEN (B.P. 281,510, 19.2.27).—Reaction vessels for use in the denitrating and nitrogen oxide absorption processes in the production of sulphuric acid have a horizontal cross-section larger than 50m.², and are filled with acid-resisting packing having an average diameter of 40 mm. The use of such enclosures gives a more steady state of thermal equilibrium inside the plant. [Stat. ref. to B.P. 249,914.]

W. G. CAREY.

Chambers for the manufacture of sulphuric acid. M. COLOMBO Y MANNI (B.P. 270,661, 31.3.27. Sp., 6.5.26).—Each chamber is of parallelepiped, rectangular, or truncated pyramid shape with a rectangular or polygonal base, and the upper part is provided with one or more sections of trapezoidal shape of the width of the chamber but of any suitable breadth and depth. The portions of the chamber at the top are connected by horizontal or inclined pipes, and are themselves connected by pipes communicating at the bottom with the top of the chamber, whilst the upper and lower portions of the chamber are connected by external pipes which may be cooled with water. All the pipes are provided with contraction and expansion cones, and may be smooth, corrugated, or provided with fins to increase the cooling surface.

W. G. CAREY.

Manufacture of nitric acid. J. H. SHAPLEIGH and C. A. BIGELOW, Assrs. to HERCULES POWDER CO. (U.S.P. 1,653,023, 20.12.27. Appl., 5.8.25).—Sodium nitrate, sulphuric acid not substantially in excess of the amount required to produce nitric acid and normal sodium sulphate, and sufficient sodium sulphate to prevent the conversion of sodium sulphate into the acid salt are fed continuously into a suitable horizontal furnace, the substances being agitated during their travel through the furnace. The temperature is so regulated as to produce gases condensing to strong nitric acid near the feed end and yielding weak nitric acid at the discharge end of the furnace.

W. G. CAREY.

[Apparatus for the production of] phosphoric acid. C. COTTE (F.P. 621,074, 3.9.26).—Phosphorus is charged through an adjustable opening leading from a supply vessel into the top of a combustion chamber, and falls on to a block of resistant material at the bottom of the chamber. A mixing chamber is attached to the combustion chamber.

L. A. COLES.

Purification of phosphoric acid. E. F. PEVERE and G. A. HENDRIE, Assrs. to PHOSPHORUS HYDROGEN Co. (U.S.P. 1,648,146, 8.11.27. Appl., 5.11.26).—Crude phosphoric acid is heated with coke and phosphate rock or tricalcium phosphate to form calcium metaphosphate, which reacts with the coke to give tricalcium phosphate, phosphorus, and carbon monoxide. The phosphorus is treated with steam as described in G.P. 406,411 (B., 1925, 242) to yield pure phosphorus pentoxide and hydrogen. T. S. WHEELER.

Separation of complex phosphotungstomolybdic acids. I. G. FARBENIND. A.-G., Assees. of E. HARTMANN (G.P. 445,151, 24.12.24).—The sodium salt of the acid, $3\text{H}_2\text{O}, \text{P}_2\text{O}_5, 24(\text{WO}_3 + \text{MoO}_3)$, is crystallised from a solution containing about 12–18% ($\text{WO}_3 + \text{MoO}_3$), and the acid, $3\text{H}_2\text{O}, \text{P}_2\text{O}_5, 18(\text{WO}_3 + \text{MoO}_3)$ is salted out in the usual manner from the residual solution. L. A. COLES.

Production of acids and alkali solutions from salts by electrolysis. WÜRTTEMBERGISCHES STATISTISCHES LANDESAMT, Assees. of F. GAISSES (G.P. 444,796, 24.2.26).—Acids and alkalis formed by the electrolysis of salt solutions are collected separately in apparatus comprising three or more cells provided with diaphragms; e.g., sulphuric acid is obtained from gypsum or anhydrite by decomposing it with an alkali carbonate and electrolysing the alkali sulphate formed in apparatus comprising three cells. L. A. COLES.

Production of sodium hydroxide, chlorine or hydrochloric acid, and ammonium chloride in gas-works or coke-oven plants. R. MAND (G.P. 426,389, 29.1.24).—The waste heat of the chimney gases is used for concentrating sodium hydroxide solution obtained by the electrolysis of sodium chloride solution, and the chlorine formed during electrolysis is used for the production of chlorobenzene, or is combined with the electrolytic hydrogen to yield hydrochloric acid, which is used for the production of ammonium chloride from the ammoniacal liquor. L. A. COLES.

Neutralisation of [crude] ammonium sulphate crystals. W. DEMANN (B.P. 255,876, 21.7.26. Ger., 24.7.25).—Acid ammonium sulphate crystals are stirred with mother-liquor freed from iron by ammonia solution, or gas liquor containing hydrogen sulphide, and made alkaline with ammonium or sodium carbonate or bicarbonate. Pyridine and ammonia are recovered in a separate apparatus, or pyridine vapour free from ammonia is obtained by maintaining the working temperature of the neutraliser below the dissociation temperature of ammonium carbonate or bicarbonate and maintaining a proper concentration of alkali in the mother-liquor. W. G. CAREY.

Saturators for the manufacture of crystalline salts. (Mrs.) E. M. WEYMAN and R. P. WALLIS, Exors. of G. WEYMAN (B.P. 281,465, 18.11.26).—Liquid to be reacted on by a gas is admitted to the saturator in a number of small streams from a gutter or pipe into a space formed between the saturator walls and a partition, and the gas enters through a pipe fitted with nozzles of various sizes disposed to produce a swirling motion and an upward and downward movement of the gas in the liquid. So that partially formed crystals may develop,

the saturator has a large conical bottom space flanged at the top to support the gas inlet pipe. W. G. CAREY.

Washing and drying gelatinous substances [e.g., silica gel]. I. G. FARBENIND. A.-G., Assees. of W. J. MÜLLER, J. DRUCKER, and H. CARSTENS (G.P. 444,965, 3.11.25).—Silica gel is prepared by causing the sol to set to a jelly in thin sheets on a frame-work, and these are subjected to a suitable washing and drying process. A. R. POWELL.

Preparation of highly active porous silica gel. I. G. FARBENIND. A.-G., Assees. of F. STÖWENER (G.P. 444,914, 20.12.25).—A silica gel prepared from a feebly acid sol containing at least 50 g./litre of silica is washed thoroughly free from acid by means of a wash-water the p_{H} value of which is adjusted between 7 and 10 by the addition of a suitable electrolyte. A. R. POWELL.

Preparation of a base-exchange material from gels. F. K. LINDSAY, Assr. to ARIZONA MINERALS CORP. (U.S.P. 1,646,297, 18.10.27. Appl., 26.6.26).—The gel formed by the interaction of sodium silicate and aluminate solutions in presence of sand is subdivided into a number of blocks before hardening is complete. These blocks are dried by exposure in open air. T. S. WHEELER.

Production of colloidal calcium carbonate. T. SHIRAIISHI (U.S.P. 1,654,099, 27.12.27. Appl., 3.8.26. Japan, 25.12.24).—To freshly prepared milk of lime, after treatment at 15° or less with carbon dioxide, a protective colloid is added, and the product is evaporated. H. ROYAL-DAWSON.

Purification and solidification of aluminium chloride. C. W. HUMPHREY and D. S. MCKITTRICK, Assrs. to C. W. HUMPHREY and H. I. LEA (U.S.P. 1,645,142—4, 11.10.27. Appl., [A] 29.5.23, [B, c] 31.5.23. Renewed [A] 18.8.27, [B, c] 6.11.26).—(A) Aluminium chloride is heated under pressure at 200–300° in one of two chambers of a retort, until liquefaction is complete. The second chamber is then cooled so that the chloride distils into it, and passes through the liquid form into a solid cake free from impurities. (B) Aluminium chloride containing ferric chloride is heated with aluminium under pressure as in (A) until the ferric chloride and aluminium have been converted into aluminium chloride and iron. The aluminium chloride is then distilled from the iron as described in (A). (C) An apparatus for performing the process described in (A) comprises a furnace in which is a retort containing two chambers. The retort can be moved so that one chamber is brought outside the furnace. T. S. WHEELER.

Manufacture of anhydrous chlorides and hydrogen chloride. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 281,491, 7.1.27).—Water-gas is treated with the equivalent amount of chlorine for the carbon monoxide and hydrogen, and the mixture, which evolves heat during the gaseous reaction, is led in counter-flow to the metallic oxide in a shaft or rotary furnace. Volatile chlorides, e.g., aluminium chloride, are deposited on cooling from the hydrogen chloride and carbon dioxide formed, from which the chlorides may be recovered completely by electrical means or by washing them with concentrated hydrochloric acid. Non-volatile

chlorides, *e.g.*, chromium chloride, are recovered from the residues. W. G. CAREY.

Manufacture of anhydrous salts of fatty acids. H. VON HOCHSTETTER, Assr. to HOLZVERKOHLLUNGS-IND. A.-G. (U.S.P. 1,645,265, 11.10.27. Appl., 7.12.26. Ger., 18.12.25).—Anhydrous ethyl acetate or a homologue is agitated with anhydrous sodium or other metal hydroxide dissolved in absolute alcohol.

T. S. WHEELER.

Manufacture of anhydrous fatty acid salts. H. VON HOCHSTETTER, Assr. to HOLZVERKOHLLUNGS-IND. A.-G. (U.S.P. 1,648,516, 8.11.27. Appl., 8.12.26. Ger., 18.12.25).—An anhydrous lower fatty acid ester, *e.g.*, ethyl acetate, is vigorously agitated at ordinary temperature with pulverised anhydrous alkali hydroxides.

T. S. WHEELER.

Purification of solutions of zinc salts. A. VOHL & Co., A.-G. (B.P. 263,809, 23.12.26. Ger., 24.12.25).—Waste liquors from the manufacture of parchment paper, artificial silk, etc. containing cellulose slimes and zinc salts are purified by first removing iron by oxidation with chlorine or peroxides in the presence of basic metal compounds corresponding to the metal ions of the solution to be purified, and, after filtration, destroying organic constituents before or during evaporation by oxidising agents, *e.g.*, chloric acid or chlorates, nitric acid, or permanganates. Zinc chlorate may be used to prevent the introduction of fresh ions, or barium chlorate, the barium being subsequently removed as barium sulphate. Any remaining traces of iron are rendered colourless by zinc and acid.

W. G. CAREY.

Production of metals and their compounds free from phosphorus. OTAVI MINEN- U. EISENBAHN- GES. (G.P. 444,862, 14.1.23).—Metal salt solutions for use in the production of metals or their compounds are freed from phosphorus by the addition of solutions containing zinc salts.

L. A. COLES.

Oxidation of metallic solutions. L. F. CLARK (U.S.P. 1,649,152, 15.11.27. Appl., 2.11.22).—The preliminary oxidising process described in U.S.P. 1,503,229 (B., 1924, 875) is claimed.

T. S. WHEELER.

Production of phosphorus trioxide. L. WOLF (G.P. 444,664, 4.9.26).—Phosphorus is burnt in oxygen or in a mixture of it with small quantities of inert gases, and the phosphorus trioxide formed is cooled immediately below its decomposition temperature, the velocity of the gas mixture being reduced and the reaction being effected under reduced pressure. The apparatus is constructed of material not affected by rapid alterations in temperature, *e.g.*, of quartz, porcelain, or metal.

L. A. COLES.

Manufacture of lead tetraethyl. H. W. DAUDT; A. E. PARMELEE, and W. S. CALCOTT, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,645,375, 11.10.27. Appl., 2.11.25).—In the manufacture of lead tetraethyl by the interaction of ethyl chloride and sodium-lead alloy, it is of advantage to add to the reaction mixture before distillation of the required product a substance which will form an insoluble film on the surface of the lead residues, *e.g.*, glue, benzene, sodium sulphide or sulphate. Agitation of the mass during the distillation is thus facilitated.

T. S. WHEELER.

Manufacture of lead tetra-alkyl. K. P. MONROE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,645,389 and 1,645,390, 11.10.27. Appl., [A] 23.10.22, [B] 19.4.24).—(A) In the preparation of lead tetra-alkyl by the action of alkyl bromide on sodium-lead alloy in presence of pyridine or similar catalyst, it is of advantage to treat the reaction mixture with saturated magnesium chloride or potassium carbonate solution rather than with water as in the usual process. (B) The process claimed in (A) is modified by the addition of an alcohol (1%) to the alkyl bromide employed.

T. S. WHEELER.

Preparation of pyrophosphates. W. H. DICKERSON, Assr. to INDUSTRIAL WASTE PRODUCTS CORP. (U.S.P. 1,654,283, 27.12.27. Appl., 15.11.23).—Particles of sodium hydrogen phosphate are heated while in suspension and motion.

II. ROYAL-DAWSON.

Production of carbonic acid gas. B. LUNDIN (B.P. 278,304, 25.11.26. Swed., 28.9.26. Cf. B.P. 268,478; B., 1928, 14).—A vessel containing solid sodium bicarbonate is disposed centrally above a tank containing a solution of sodium bisulphate; a rotating shaft which passes through both vessels stirs the liquid in the lower tank and feeds bicarbonate from the upper vessel into the lower, which is kept at about 33°. The saturated solution of sodium sulphate so formed is withdrawn, cooled to 15°, and the crystals recovered. The pressure of the evolved gas is regulated by a pump or compressor, and air is expelled on starting the process by the introduction of carbon dioxide or steam, whilst at the end absorbed carbon dioxide is withdrawn from the solution by suction.

W. G. CAREY.

Dehydration of ammonia vapours. C. COOPER and D. M. HENSHAW, Assrs. to W. C. HOLMES & Co., LTD. (U.S.P. 1,654,863, 3.1.28. Appl., 5.2.27. U.K., 16.12.25).—See B.P. 272,970; B., 1927, 652.

Preparation of titanous oxide from titanous ores. J. D'ANS and F. SOMMER (U.S.P. 1,655,940, 10.1.28. Appl., 28.4.23. Ger., 14.12.21).—See B.P. 214,483; B., 1924, 511.

Manufacture of pure zirconium sulphate. H. TRAPP (U.S.P. 1,648,569, 8.11.27. Appl., 2.10.24. Ger., 25.9.23).—See B.P., 222,486; B., 1925, 630.

Apparatus for roasting pyrites (G.P. 444,991).—See I. **Acetic and formic acids** (B.P. 291,827).—See III.

VIII.—GLASS; CERAMICS.

Brilliant gold. CHEMNITIUS.—See X.

PATENTS.

Tunnel kiln. W. L. HANLEY, JUN. (U.S.P. 1,653,174, 20.12.27. Appl., 30.8.26. Renewed 17.5.27).—A central zone of the tunnel is provided with direct firing means discharging its combustion products into the upper portion of the goods space, and spaced ports in the lower portion of this space communicate with eduction passages. Passages are also provided in line with the ports for viewing the goods in the lower portion from the exterior of the kiln.

H. HOLMES.

Working of quartz. E. R. BERRY and P. K. DEVERS, Assrs. to GEN. ELECTRIC CO. (U.S.P. 1,645,086, 11.10.27. Appl., 20.9.23).—One end of a silica rod is heated and

expanded mechanically by a former and by gas pressure into a tube, the size of which is fixed by a mould. As formation proceeds, the rod progressively advances into the apparatus. T. S. WHEELER.

Production of high-grade vitreous silica. H. L. WATSON, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,645,080, 11.10.27. Appl., 24.6.26).—Silica treated as described in U.S.P. 1,536,821 (B., 1925, 548) is heated for 10 min. at atmospheric pressure at 2300°. T. S. WHEELER.

Manufacture of magnesite refractories. U.S. METALS REFINING Co., Assees. of A. MARKS (B.P. 276,016, 6.5.27. U.S., 8.6.26. Addn. to B.P. 244,391; B., 1926, 586).—Linseed oil or similar siccative oil is rendered more viscous before or after admixture with the powdered magnesite by heating or drying. The mixture may also be conditioned before moulding by being kept, or after moulding. W. G. CAREY.

Formation of refractory crucibles. H. T. REEVE, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,648,149, 8.11.27. Appl., 24.3.26).—A paste of a refractory oxide is applied in a series of layers to a form coated with a paper separator, so that the crucible can be easily removed after drying; it is then fired. T. S. WHEELER.

Apparatus for the production of artificial jewels or precious stones. L. SCHMIDT, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,653,022, 20.12.27. Appl., 10.10.25. Ger., 12.7.24).—A gas flame flows round a pin which projects inside a vessel of refractory material and separately suspended particles of powdered aluminium oxide are applied to the point of the pin by compressed gas from a vessel containing powdered aluminium oxide kept in suspension by a rotary device in the vessel. W. G. CAREY.

Manufacture of a refractory article. A. J. JACKMAN, Assr. to VESUVIUS CRUCIBLE Co. (Re-issue 16,845, 3.1.28, of U.S.P. 1,577,124, 16.3.26).—See B., 1926, 542.

Production of molten liquid, coats of enamel, glass, etc. by spraying upon metal, stone, or other surfaces or bodies. A. PAHL (B.P. 281,885, 7.1.27).

Leer construction. PITTSBURGH PLATE GLASS Co., Assees. of H. S. HEICHERT (B.P. 275,169, 18.5.27. U.S., 29.7.26).

IX.—BUILDING MATERIALS.

Use of volcanic ash for the preparation of hydraulic cement. H. BRINTZINGER and W. BRINTZINGER (Z. anorg. Chem., 1927, 168, 93—95).—Cement freshly prepared from Guatemalan volcanic ash is of considerable mechanical strength, but on ageing in presence of air and moisture, both the free and the combined sulphur in the ash are oxidised to sulphate. Calcium and aluminium sulphates are thus formed, with a sufficient increase of volume to lower considerably the strength of the cement. Volcanic ash containing sulphur is therefore not a probable competitor of Portland cement.

H. F. GILLBE.

Measurement of moisture movements in building materials. F. L. BARROW (J. Sci. Instr., 1927, 4, 475—480).—Two extensometers for use in measuring the expansion or contraction of building materials under varying moisture conditions are described. Both instru-

ments are mechanical in principle and consist essentially of a lever mechanism, with an optical arm, to magnify the actual movement of the specimen. Movements may be recorded autographically. Both instruments may be used to record length changes with changing humidity of the surrounding air or on immersion in water.

J. S. CARTER.

PATENTS.

Rotary kiln and cooler [for cement manufacture]. P. T. LINDHARD, Assr. to F. L. SMIDTH & Co. (U.S.P. 1,653,050, 20.12.27. Appl., 19.11.25).—The discharge end of the kiln supports an inner and an outer annular series of cooling drums external to it. The clinker passes from the kiln through the inner series and thence into one end of the outer series, and is finally discharged from the other end. H. HOLMES.

Separation of fine sand from sludge. O. N. RIKOF (B.P. 278,468, 22.7.26).—A pipe for delivering clean water is led through the centre of a sludge pipe and opens in a downward direction over the outlet in the bottom of a hopper to prevent accumulation of sand near the outlet and to ensure a steady and uniform discharge. A diffuser in the form of a horizontal shield is fixed to the bottom of the pipe and additional water is supplied by radially disposed pipes immediately above the outlet. The upward flow of water causes the lighter particles to overflow at the top of the hopper.

W. G. CAREY.

Manufacture of a waterproof plastic Portland cement composition. H. V. WELCH, Assr. to INTERNAT. PRECIPITATION Co. (U.S.P. 1,644,964—5, 11.10.27. Appl., [A] 26.2.24, [B] 5.1.25).—Portland cement is mixed with (A) bituminous kieselguhr or (B) oil shale.

T. S. WHEELER.

Prepared fireproof mortar. R. L. TAYLOR, Assr. to TOUPET-TAYLOR ENGINEERING Co. (U.S.P. 1,645,030, 11.10.27. Appl., 5.1.23. Renewed 13.8.27).—An intimate mixture of ferromanganese slag, the residue obtained by burning coal-mine slate and waste ("red dog"), and a small proportion of cement is claimed.

T. S. WHEELER.

Production of fire-resistant fibrous materials. A. WINOGRADOFF, Assr. to IMPROVED OFFICE PARTITION Co. (U.S.P. 1,645,172—3, 11.10.27. Appl., 22.12.26).—Wood impregnated with a solution containing magnesium sulphate and potassium hydrogen carbonate is heated at 60° to precipitate magnesium carbonate in the fibre.

T. S. WHEELER.

Manufacture of artificial stone or plaster. W. A. OAKLEY (B.P. 281,757, 6.9.26).—A hot aqueous solution of a metallic chloride capable of forming an oxychloride with magnesia, *e.g.*, magnesium, ferric, or calcium chlorides, containing also 5—10% of an alkali silicate, is mixed with finely-divided magnesia or calcined magnesite and a finely-divided filler, *e.g.*, metallic oxides, siliceous or aluminous clay, calcareous earth, etc. If the filler be a calcareous earth or alkaline clay, an alum or other metallic sulphate is added to the liquid metallic chloride instead of the silicate, and the product so obtained with the filler is set, powdered, and mixed with about double the amount of magnesite to form a cement.

W. G. CAREY.

Pitch compositions. PRODORITE, LTD., Assees. of CONTINENTALE PRODORIT A.-G. (B.P. 256,640, 6.8.26. Ger., 6.8.25).—Hard pitch, which does not soften below 40°, as indicated by the Brinell hardness test (cf. B.P. 201,650 and 237,010; B., 1923, 977 A; 1925, 762), is incorporated with 10–15% of asbestos fibre or flakes, preferably with the addition of stone dust, sand, etc. W. G. CAREY.

Asphalt or bitumen substitute. F. G. RENO, Assr. to AUSTRALIAN BITUMINOUS COMPOUNDS, LTD. (U.S.P. 1,649,545, 15.11.27. Appl., 7.9.27. Austral., 12.5.26).—A filling material is incorporated with a basic material consisting of oil-bearing shale and tar.

A. C. MONKHOUSE.

Road making. J. RADCLIFFE (U.S.P. 1,655,240, 3.1.28. Appl., 26.12.24. U.K., 2.1.24).—See B.P. 236,050; B., 1925, 720.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Calculation of the carbon consumed in direct reduction [of the iron ore] in the blast furnace. E. MAURER (Arch. Eisenhüttenw., 1927, 1, 331–337; Stahl u. Eisen, 1928, 48, 7–8).—A review of the work of earlier investigators, in which an attempt has been made to explain the discrepancies in the various formulæ proposed. The most probable cause of the disagreement in the results obtained by the use of these formulæ is an error in the calculation of the quantity of air used, the figures for which based on the nitrogen content of the flue gases do not agree with those based on the oxygen content.

A. R. POWELL.

Use of nickel in cast iron and steel. J. GALIBOURG (Rev. Mét., 1927, 24, 730–739).—Addition of nickel to molten iron for castings favours the transformation cementite \rightarrow graphite \rightarrow ferrite, and gives a more homogeneous distribution of graphite. Examples of the composition of a number of cylinders for various purposes and of nickel-steel castings are given. C. A. KING.

Use of nickel in armaments. G. CHARPY (Rev. Mét., 1927, 24, 715–721).—A review of the introduction and progress of armouring in naval warfare, including the discovery of differential tempering of nickel alloy steel plates.

C. A. KING.

Use of nickel in automobile and aviation industries. L. GUILLET (Rev. Mét., 1927, 24, 722–729).—A review of alloy steels and their properties in use in motor and aviation industries. There are few special steels used in machine construction which do not contain nickel, the addition of which improves the mechanical properties and renders alloy steels more amenable to special treatments.

C. A. KING.

Non-magnetic alloys of nickel. J. COURNOT (Rev. Mét., 1927, 24, 740–763).—Equilibrium diagrams and the physical properties of a number of non-magnetic alloys of nickel are given, including nickel with copper, cobalt, manganese, molybdenum, zinc, cadmium, tin, lead, aluminium, magnesium, silver, gold, platinum, also brasses, bronzes, and other triple alloys.

C. A. KING.

Corrosion of nickel alloys. A. PORTEVIN (Rev. Mét., 1927, 24, 697–714).—After commenting on the difficulty of arriving at conclusions from results of corrosion tests and stating that there is no generalisation in the resistance of alloys to corrosion, nickel is regarded as the one metal which is particularly useful in resistant alloys, regarding these from the points of view of malleability, resistance to chemical attack, and oxidation on heating, and the commercial aspect of cost.

C. A. KING.

Properties of electrolytic nickel. B. BOGITCH (Compt. rend., 1927, 185, 1467–1469).—The compositions and physical and mechanical properties of a number of samples of electrolytic nickel are tabulated and shown to be dependent on the method of preparation. The rates of corrosion of the metals by dilute hydrochloric, sulphuric, or nitric acids at 70°, or in air, follow the same order as their apparent densities, the heavier metals being the least attacked. Reheating lowers the hardness and increases the rate of corrosion. J. GRANT.

Causes of failure in heat-resisting alloys. R. SUTTON (Trans. Amer. Soc. Steel Treat., 1927, 12, 221–234).—Carburising boxes containing Ni 60%, Cr 18%, and Fe 15% are preferred. CHEMICAL ABSTRACTS.

Physical characteristics of commercial copper-zinc alloys. W. H. BASSETT and C. H. DAVIS (Amer. Inst. Min. Met. Eng. Tech. Pub., 1927, No. 26, 16 pp.).—Tensile, alternate bending, and hardness tests were performed on copper-zinc alloys, and the effect of annealing on the structure and grain size was investigated. Impurities, e.g., iron, lower the grain size and increase the hardness. CHEMICAL ABSTRACTS.

Heat treatment of aluminium-silicon alloys. R. S. ARCHER, L. W. KEMPF, and D. B. HOBBS (Amer. Inst. Min. Met. Eng. Tech. Pub., 1927, No. 23, 30 pp.).—Quenching from 565°, as compared with slow cooling, produces increased hardness and decreased plasticity and elongation. Ageing at elevated temperatures increases the hardness. Spheroidising and growth of the silicon particles are marked above 565°.

CHEMICAL ABSTRACTS.

Equilibrium relations in aluminium-silicon and aluminium-iron-silicon alloys of high purity. E. H. DIX, JUN., and A. C. HEATH, JUN. (Amer. Inst. Min. Met. Eng. Tech. Pub., 1927, No. 30, 31 pp.).—The aluminium-silicon eutectic contains 11.82% Si. The position of the solidus at the aluminium end was determined. Examination of alloys prepared at the intersection of Al-FeSi₂ and of FeAl₃-Si on the phase diagram indicated that the line Al-FeSi₂ and FeAl₃-Si does not exist. The compound FeAl₃ exists.

CHEMICAL ABSTRACTS.

Choice and co-ordination of electrolytic, gravimetric, and volumetric methods for the analysis of white metals, bronzes, and ordinary and special brasses. A. TRUCCO (Annali Chim. Appl., 1927, 17, 570–589).—Methods are given for the complete analysis of white metals, white metals rich in lead, white metals composed mainly of tin and antimony, bronzes, and brasses.

T. H. POPE.

Treatment of complex raw speiss. W. F. KAISER (Continental Met. Chem. Eng., 1927, 2, 195–198, 238–

242).—A discussion of the electrolytic method of treatment, using basic or acid electrolytes. Direct electrolysis, after concentration, is permissible if copper, nickel, cobalt, and some arsenic are the chief metallic constituents.

CHEMICAL ABSTRACTS.

Effect of boiling orange-juice on various metals and alloys. A. L. BLOUNT and H. S. BAILEY (Trans. Amer. Inst. Chem. Eng., 1926, 18, 139—148).—A number of metals are classified in order of resistance to the corrosion.

CHEMICAL ABSTRACTS.

Brilliant gold. F. CHEMNITUS (J. pr. Chem., 1927, [ii], 117, 245—261).—A detailed account of the various substances used in the preparation of brilliant gold for pottery decoration.

H. BURTON.

Metallurgy of high-frequency induction furnaces. F. WEVER and G. HINDRICH (Arch. Eisenhüttenw., 1927, 1, 345—355; Stahl u. Eisen, 1928, 48, 11—13).—Tests with 30-, 50-, and 100-kw. high-frequency induction furnaces using magnesite-tar, fireclay, and fireclay-graphite crucibles showed that these furnaces are especially suitable for the manufacture of high-grade cutlery steel and high-speed tool steel. The current consumption in the largest furnace is 880 kw.-hr./ton starting from cold, and 1265 kw.-hr./ton in the smallest furnace. Soft iron and chromium-steel prepared in an acid crucible were free from hot-shortness and their mechanical properties were equal or superior to those of similar steels produced in an acid, open-hearth furnace. In a basic crucible the sulphur content is rapidly reduced to 0.008%, and the loss of tungsten and vanadium is relatively small.

A. R. POWELL.

PATENTS.

Furnace for roasting ore. M. McGUINNESS (B.P. 281,837, 3.11.26).—An ore-roasting furnace, particularly suitable for treating cinnabar, is provided with means for aiding the passage of the ore through the furnace, which is heated by electric resistance bars placed in any suitable positions. The inlet and exit for solid residue of the furnace are preferably water-sealed to prevent the escape of mercury vapour.

C. A. KING.

Roasting [of sulphide ores]. J. B. READ and M. F. COOLBAUGH, ASSS. to COMPLEX ORES RECOVERIES CO. (U.S.P. 1,644,692, 11.10.27. Appl., 29.7.25).—A furnace for performing the process of B.P. 200,852 (B., 1923, 1077A) contains a number of superposed hearths, each fitted with rotating rakes. All but a small portion of the air necessary for oxidation is introduced at about the third hearth from the top and travels downwards with the material under treatment; the remainder is circulated by a fan up through the first two hearths and back to the third to preheat the entering material.

T. S. WHEELER.

[Ore] flotation machine. F. DE MIER, ASSR. to W. A. BUTCHART (U.S.P. 1,646,351, 18.10.27. Appl., 29.12.25).—The machine comprises a bladed cylinder rotating in a casing, in which are openings to permit of removal of the froth.

T. S. WHEELER.

Pickling, annealing, and otherwise treating metal sheets. J. C. DAVIES. From W. E. WATKINS (B.P. [A] 281,349, [B] 281,575, [C, D] 281,578—9, 16.6.26).—(A) Metal sheets are arched, pickled, washed, decrowned, dried, arched again, annealed, decrowned, cooled, arched

again, pickled, decrowned, and finally straightened by means of suitable automatic machinery. (B) For the pickling operation the sheets are arched transversely and passed, by means of suitable conveyors, through a steam-heated bath of dilute acid, thence through a washing vessel, where they are washed by hot-water sprays and from which they pass over a series of gas flames for drying, and finally through a decrowning press. (C) In the annealing operation the crown of the sheet is heated first to relieve the internal stress in that portion, and the whole sheet is then passed to the annealing furnace, which is maintained at 880° by means of hot, non-oxidising gases under slight pressure to prevent ingress of air. (D) The hot sheets from the annealing furnace are transferred to a cooling chamber through which the waste inert gases from the annealing furnace are passed to cool the sheets to such a temperature that they will not oxidise on exposure to the air. The chamber is provided with a cooling system of pipes through which cold water is passed.

A. R. POWELL.

[Blast furnace for] smelting of iron ores. Y. MURAKAMI (F.P. 621,588, 16.9.26. Japan, 28.9.25).—An iron blast furnace is provided with a lower crucible chamber which can be closed by means of a rotating device. Just above the latter are arranged tuyères for removing part of the gases, charging doors for introducing fuel, and pipes for the introduction of air and, if required, cooling gases. The process of combustion is so regulated that the ore is reduced in the upper zones by the partially burnt gases, too rapid melting is avoided, and the waste gases pass through the mass without interference. By closing the crucible chamber the molten iron can be subjected to a purification process before removal from the furnace.

A. R. POWELL.

Removal of phosphates from iron ores. L. P. BASSET (F.P. 619,218, 24.11.25).—The ore is leached with very dilute hydrochloric or sulphuric acid at the ordinary temperature, and the solution of phosphoric acid so obtained is utilised for the preparation of fertilisers. Coarse ores are roasted before treatment to render them pervious to the acid. The purified ore is washed thoroughly on the counter-current principle previous to smelting.

A. R. POWELL.

Manufacture of pure iron. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 281,963, 25.6.27. Addn. to B.P. 262,938; B., 1927, 144).—Iron carbonyl in the undiluted liquid or vaporous condition is passed through the heated liquid or melt at ordinary or under increased pressure, or brought into contact with the surface of the liquid or melt by blowing the vapour or spraying the liquid on to the agitated surface. Organic liquids of high b.p., melted salts, or metals may be used, and the working temperature should be between 100° and 400° or above 1000° in order to prevent extensive deposition of carbon as the result of the decomposition of carbon monoxide.

M. E. NOTTAGE.

Tempering of steel. J. F. EDWARDS (U.S.P. 1,648,044, 8.11.27. Appl., 29.10.25).—An aqueous quenching bath contains iodine, sodium chloride, and glycerol.

T. S. WHEELER.

Coating of iron articles with tin and zinc. T. LIBAN (B.P. 281,357, 20.7.26).—Articles which have been

cleaned mechanically or chemically are introduced into a bath of molten lead through a substantial covering layer of a mixture of fluorine compounds with zinc chloride or zinc ammonium chloride, and withdrawn through molten zinc (which may contain aluminium) floating on the surface of the lead at the exit end of the bath.

C. A. KING.

Decomposition of chromite. I. G. FARBENIND. A.-G., Assees. of C. MÜLLER, L. SCHLECHT, and A. CURS (G.P. 444,798, 21.10.24).—Chromite is first reduced in hydrogen or in a mixture of gases containing hydrogen and the product is heated above 200° with a slight deficiency of sulphuric acid in a closed vessel lined with hard lead containing preferably 3% Sb.

A. R. POWELL.

Manufacture of [high-percentage] ferrophosphorus. J. WARNER (U.S.P. 1,646,268, 18.10.27. Appl., 16.6.27; cf. U.S.P. 1,475,976; B., 1924, 60).—Coke, phosphate rock, and iron are smelted in a blast furnace to give ferrophosphorus containing 10–16% P. This is recharged to the furnace with iron, coke, and a restricted amount of phosphate rock. A product containing over 20% P is obtained.

T. S. WHEELER.

Treatment of ores of copper and other metals. F. DIETZSCH (B.P. 281,741, 7.7.26).—Prior to leaching, ores are roasted with a sulphate of an alkali or alkaline-earth metal, not exceeding 2% of the weight of ore.

C. A. KING.

Hardening of copper and copper alloys. O. W. GUSTAFSON (B.P. 281,799, 21.9.26).—The metal is plunged into a bath formed by an inorganic acid, e.g., nitric acid, and a neutral alkaline salt, e.g., ammonium chloride or borax, both metal and bath being at ordinary room temperature.

M. E. NOTTAGE.

Roasting of zinc sulphide ores. SOC. GÉN. MÉTALLURGIQUE DE HOBOKEN (B.P. 271,877, 23.5.27. Fr., 27.5.26).—The raw ore containing sulphur is mixed with a natural ore which does not contain sulphur, such as oxide or carbonate ore, or with a sulphur-containing ore which has been already sintered, and the mixture is treated direct in a roasting blast apparatus wherein it is completely desulphurised and subsequently reduced. By this means ores in a fine state of division, which would otherwise cause trouble in the subsequent operations of reduction and distillation in retorts, may be obtained in a well-sintered condition.

M. E. NOTTAGE.

Reduction of zinc ores. H. WITTEK (B.P. 279,370, 20.11.26. Ger., 23.10.26).—The ores are reduced by heating them for about 1 hr. with carbide in a current of nitrogen at a temperature of 1100–1150°, the rate of reduction being regulated by controlling the supply of nitrogen.

M. E. NOTTAGE.

Aluminium alloy. H. C. HALL and T. F. BRADBURY (B.P. 281,912, 17.3.27).—An alloy of aluminium contains 0.5–0.4% Cr, 0.2–1.5% Ni, 0.1–1% Mg, up to 0.5% Sb, up to 0.5% Ti, 0.2–1.5% Si, 0.7–1.5% Fe, and 2–5% Cu.

C. A. KING.

[Malleable] alloys. T. H. KELLY (B.P. 281,950, 31.5.27).—Malleable alloys of nickel, copper, and iron are produced by melting the metals rapidly in the non-oxidising atmosphere of an electric furnace. Suitable

proportions are 20–40 pts. of nickel, 20–40 pts. of copper, and 40–60 pts. of iron. To give greater hardness, up to 20% of the iron may be replaced by chromium.

C. A. KING.

Magnetic alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 281,763, 8.9. and 17.12.26).—Magnetic alloys contain 10–17% (12.5–13.5%) Fe, 1.5–4% (1.5–2%) Si, 1–4% (2–2.5%) of a fourth element, e.g., chromium, tungsten, vanadium, or molybdenum, and the balance nickel.

C. A. KING.

Production of lamellar metallic deposits. W. A. F. PFANHAUSER (B.P. 274,426, 11.5.27. Ger., 14.7.26).—Lamellar metallic deposits, e.g., nickel, are produced on an endless belt acting as cathode and passing repeatedly through different solutions to separate the cumulative layers of metal. Thus the band might pass alternately through nickel and copper solutions, or through a nickel solution and a dilute solution of wax, the intermediate layers in each case being dissolved, leaving nickel flakes.

C. A. KING.

Treatment of articles [deposition of metal on articles] in metallic baths. N. J. BARBIER (U.S.P. 1,654,910, 3.1.28. Appl., 28.10.25. Fr., 31.10.24).—The portion of molten metal immediately surrounding the article immersed in the bath is heated to a temperature not less than that of the remainder of the molten metal by passing an electric current between the immersed article and the molten metal used as electrodes.

J. S. G. THOMAS.

Open-hearth furnace. F. B. MCKUNE, Assr. to OPEN HEARTH COMBUSTION Co. (Re-issue 16,837, 27.12.27, of U.S.P. 1,339,855, 11.5.20).—See B., 1920, 493 A.

Arrangement of the gas and air passages in reverberatory furnaces. H. MOLL (U.S.P. 1,653,405, 20.12.27. Appl., 6.5.22. Ger., 20.5.21).—See B.P. 177,812; B., 1923, 893 A.

Coating [of pipes etc.]. B. TALBOT, Assr. to TALBOT NON-CORROSIVE LININGS Co. (U.S.P. 1,653,746, 27.12.27. Appl., 12.5.25. U.K., 24.4.25).—See B.P. 255,546; B., 1926, 837.

Misch-metal wire. J. W. MARDEN and M. N. RICH, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,653,367, 20.12.27. Appl., 29.1.25).—See B.P. 246,860; B., 1926, 884.

Coating of materials by metal spraying. F. G. COZENS, and METALLISATION, LTD. (B.P. 282,116, 17.7.26).

Roasting of pyrites (G.P. 444,911).—See I. **Washing of ores** (B.P. 265,202).—See II. **Detonator tubes** (G.P. 443,727).—See XXII.

XI.—ELECTROTECHNICS.

Treatment of raw speiss. KAISER. **High-frequency induction furnaces.** WEVER and HINDRICHES.—See X.

PATENTS.

Electric furnace. A. E. GREENE (U.S.P. 1,654,419, 27.12.27. Appl., 23.11.22).—An outwardly-bulged, horizontal, cylindrical shell has a refractory lining and cone-shaped metal ends. A moving door member abuts against a flat end surface on one cone-shaped end, and

a doorway opens through this end and the refractory lining. J. S. G. THOMAS.

Electric [reducing] furnace. P. L. J. MIGUET, Assr. to SOC. ELECTROMÉTALLURGIQUE DE MONTRICHER (U.S.P. 1,655,324, 3.1.28. Appl., 29.9.26. Fr., 31.12.25).—Sandwiched conductors are arranged within ducts arranged outside the furnace walls, and if desired air-cooled without the air entering the furnace lining. Transformer chambers are arranged in the ducts, and the apparatus is supported independently of the furnace walls. J. S. G. THOMAS.

Material for commutator brushes. F. C. ATKINSON (U.S.P. 1,644,703, 11.10.27. Appl., 25.4.23).—Pitch is heated with cuprous or cupric oxide to yield a mixture of copper and carbon, which is powdered, moulded with a binder of copper oxide and pitch, and baked. T. S. WHEELER.

Manufacture of a composition of matter [insulating material]. M. DARRIN, Assr. to F. N. BURT Co., LTD. (U.S.P. 1,644,711, 11.10.27. Appl., 10.6.24. Renewed 11.3.27).—Cellulose is impregnated with molten sulphur containing naphthalene or triphenyl phosphate to increase its fluidity. An insulating material capable of being machined is obtained on cooling. T. S. WHEELER.

Removal of enamel from electrical conductors. W. G. HOUSEKEEPER, Assr. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,653,805, 27.12.27. Appl., 10.2.26).—Enamel formed of a solid hydrocarbon and a non-drying vegetable oil subjected to destructive distillation, and fibrous insulation, are stripped from the conductor after the enamel has been softened by treatment with monochlorobenzene, and the insulation has been impregnated with wax. J. S. G. THOMAS.

Electrolytic treatment for the cylinders of rotary printing machines to eliminate polishing and wear. H. KUPPEL and L. SIMÉANT (B.P. 282,274, 10.5.27).—Engraved cylinders are employed successively as cathodes or anodes, respectively, in an electrolytic bath consisting of 78% of water, 18% of copper sulphate, 4% of sulphuric acid (*d* 1.14), in which copper sheets are suspended. The bath is operated with a voltage not exceeding 3 volts, and the current density is maintained constant at 0.4–0.5 amp. cm.² J. S. G. THOMAS.

Electrical purification of gases. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of H. LÜBKE (G.P. 445,032, 6.2.26).—The gas is mixed with the vapour of an insulating fluid, *e.g.*, an oil, which has a dielectric constant greater than 1, and the mixture is passed through the usual electrical purification plant. A. R. POWELL.

Corrugated sheet-metal electrodes for electrical gas-purification plant. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of G. KARKUTSCH (G.P. 444,963, 14.10.24).—The electrodes consist of thin, corrugated, sheet metal strengthened by means of cross-pieces joining the crests of the corrugations and arranged at regular distances apart. A. R. POWELL.

Electric lamp [with non-sagging filament]. A. DE GRAAFF, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,644,712, 11.10.27. Appl., 17.4.23. Holl., 27.4.22).—Sagging of the filament of gas-filled lamps is inhibited by mixing

hydrogen (0.5–4%) with the argon filling, and sealing in phosphorus suboxide (P₄O) to act as a drying agent. T. S. WHEELER.

Illuminating tubes. J. BRETTMON (F.P. 617,842–3, 3.11.25).—(A) Tubes, highly evacuated or filled with gases or metallic vapours and provided with electrodes, contain a small amount of radium salt or substance containing radium. Such tubes become luminous when consuming very much less electrical energy than is the case when radium is absent. (B) Tubes of glass or similar material, having a carbon and a metallic oxide electrode, are evacuated as completely as possible. When operated with a suitable voltage, the carbon electrode becomes incandescent and the metallic oxide is decomposed so that the tube is filled with metallic vapour and carbon monoxide or dioxide. A white or coloured incandescent results. The tubes can then be operated by means of indifferent electrodes. J. S. G. THOMAS.

Photo-electric cell. WESTINGHOUSE ELECTRIC & MANUF. Co., Assees. of V. K. ZWORYKIN (B.P. 271,476, 17.5.27. U.S., 20.5.26).—The active material of a photo-electric cell consists of an alloy of metals of the alkali and alkaline-earth groups, respectively, prepared by chilling the two metals to the temperature of liquid air, then mixing the constituents *in vacuo*, and heating. J. S. G. THOMAS.

Photo-electric cell. TELEFUNKEN GES. F. DRAHTLOSE TELEGRAPHIE M.B.H., and O. VON BRONK (G.P. 443,971, 15.8.24).—An electrode, thinly coated with selenium by sputtering or sublimation, and a heated cathode are arranged in a vessel which is either highly evacuated or filled with gas. Almost the whole of the selenium through which current is passed can be exposed to illumination. Illumination of the selenium surface by the hot cathode is reduced by employing for the latter a strongly electron-emitting material, *e.g.*, an oxide or thorium filament, which is operated at such a low temperature that it produces little or no luminous effect. J. S. G. THOMAS.

Electron tube. H. A. BROWN, Assr. to UNIV. ILLINOIS (U.S.P. 1,653,544, 20.12.27. Appl., 17.7.22).—To transform thermal into electrical energy, a conductor is heated and discharges electrons through an alkali-metal vapour (*e.g.*, of potassium-sodium alloy) towards a second conductor, and an electric current is established between the two conductors by the resulting difference of potential. J. S. G. THOMAS.

Production of metallic coatings on metal electrodes used in mercury vapour rectifiers. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of M. SCHENKEL (G.P. 444,597, 7.11.24).—Iron or steel rods, prior to use in mercury-vapour rectifiers, are amalgamated by exposure in a vacuum mercury arc. The use of such electrodes prevents short-circuiting and striking-back. J. S. G. THOMAS.

Manufacture of electrodes for batteries. D. PEPPER (U.S.P. 1,653,872, 27.12.27. Appl., 1.12.24).—The active paste is compressed, while plastic, between the supporting walls of the grid and cores of readily fusible material, and the cores are removed by fusion after the paste has set. J. S. G. THOMAS.

Electrolytic cell. H. O. SIEGMUND, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,653,879, 27.12.27. Appl., 22.10.24).—Tin electrodes, carried by aluminium supports, are immersed in a film-forming electrolyte contained in a casing, the whole forming an electrolytic condenser. J. S. G. THOMAS.

Depolariser. J. T. SZEK (F.P. 619,307, 26.7.26. Belg., 5.7.26).—Aluminium chloride is used in Leclanché and similar cells in place of, or in conjunction with, a small amount of graphite and manganese dioxide as depolariser. Thus a solution of aluminium chloride and, if desired, a compound of mercury or zinc chloride is added to a paste containing rice, powdered wood charcoal, and absorbents. J. S. G. THOMAS.

Production of arc-light electrodes. L. KAHL, Assr. to RÜTGERSWERKE A.-G., ABTG. PLANIWERKE (U.S.P. 1,654,046, 27.12.27. Appl., 31.5.23. Ger., 9.6.22).—See B.P. 199,018; B., 1924, 550.

Protection of carbon or graphite electrodes in fused electrolytes. E. A. ASHCROFT, Assr. to NEW METALLURGY, LTD. (U.S.P. 1,653,605, 20.12.27. Appl., 23.2.27. U.K., 27.11.25).—See B.P. 269,749; B., 1927, 492.

Electrolytic production of acids and alkalis (G.P. 444,796).—See VH. **Magnetic alloys** (B.P. 281,763). **Metallic deposits** (B.P. 274,426).—See X.

XII.—FATS; OILS; WAXES.

Behaviour of soaps of various oils on dilution. H. B. STOCKS (Oil Fat Ind., 1927, 4, 315—319).—Hydrolysis of soaps is maximal at a dilution of 1 in 150. There is no appreciable hydrolysis of the sodium salts of fatty acids up to and including lauric acid, but above this 50% of the alkali is liberated; with castor-oil soap only 5% is liberated, since sodium ricinoleate is not largely hydrolysed. For distinguishing castor oil, 5 g. are hydrolysed under a reflux condenser with excess of alcoholic potash, phenolphthalein is added, the liquid neutralised with hydrochloric acid, and the alcohol evaporated. The residue is dissolved in water and diluted to 100 c.c.; 10 c.c. are diluted to 250 c.c. with boiling, distilled water and titrated with 0.1N-hydrochloric acid, first with phenolphthalein and then with methyl orange. With castor oil, 0.5—0.8 c.c. are required in the first titration; other fats and oils (except butter, coconut, and palm-nut fats) require 8—9 c.c.

CHEMICAL ABSTRACTS.

Behaviour of fish oils with uranium nitrate and pyrogallol. W. H. DICKHART (Oil Fat Ind., 1927, 4, 326—328).—When fish oil (3 c.c.) is heated with occasional shaking on a steam-bath for 20 min. with uranyl nitrate (10 mg.) the following colour reactions are obtained: cod-liver oil (U.S.P.), amber, greenish with transmitted light; Norwegian sperm oil, light amber, no change with transmitted light; menhaden oil, crimson; pilchard oil, light red; whale oil, light brownish-red; herring oil, sardine oil, and Newfoundland cod oil, blood-red.

CHEMICAL ABSTRACTS.

Grape seed oil. E. CARRIÈRE and BRUNET (Compt. rend., 1927, 185, 1516—1518; cf. André, A., 1922,

i, 908; 1923, i, 437).—The following acids were shown to be present in a commercial sample of grape seed oil: stearic (10%), palmitic (5%), erucic (5%), hydroxy-acids (10%), oleic (20%), and linoleic (50%).

H. BURTON.

Determination of the purity of olive oils. C. MILANI (Annali Chim. Appl., 1927, 17, 589—590; cf. B., 1927, 915).—A reagent prepared by dissolving a few drops of pyridine in a 0.5—1% solution of eosin in acetone gives no coloration with pure olive oil, a brown coloration with the commercial mixed oil, and a mauve coloration with sesamé oil. T. H. POPE.

Determination of iodine value [of oils]. E. RUPP (Apoth.-Ztg., 1927, 42, 281—282; Chem. Zentr., 1927, i, 2584).—The method of the German pharmacopœia is modified to avoid a high bromine pressure and sensitiveness to photochemical reduction. Potassium bromide (4 g.) is added to 0.2N-potassium bromate solution; after keeping in the dark for 2 or (for drying oils) 20 hrs., potassium iodide (0.5 g.) is added and the iodine titrated with 0.1N-sodium thiosulphate.

A. A. ELDRIDGE.

PATENTS.

Treatment of drying and semi-drying fats and oils. G. PETROFF (B.P. 281,896, 1.2.27).—Drying and semi-drying fats and oils mixed with not more than 15% of aromatic hydrocarbons, phenols, or hydrogenated hydrocarbons or phenols are treated with not more than 15% of concentrated sulphuric acid. After separation of the acid and removal of excess of solvent by distillation, the neutral glycerides are split in the usual way in the presence of sulpho-acids of high mol. wt. The introduction of hydrocarbons etc. effects reduction of the amount of acid used and improvement of the quality of the final product. S. S. WOOLF.

Production of neutral fat and soap from soap stock. ARTIEBOLAGET SEPARATOR (F.P. 621,112, 6.9.26. Swed., 18.11.25).—Neutral fat is separated from the stock, diluted with water if necessary, by centrifugal means, and soap is salted out of the remaining liquor.

L. A. COLES.

Flaking of soap. B. W. COLTMAN (U.S.P. 1,653,390, 20.12.27. Appl., 15.8.21).—Liquid soap is sprayed in a thin film which is then desiccated and disintegrated into flakes. H. ROYAL-DAWSON.

Oil hardening with nickel and copper formates. C. ELLIS (U.S.P. 1,645,377, 11.10.27. Appl., 1.2.24).—The presence of copper formate (10—20%) in nickel formate enables reduction of the latter by hydrogen, when suspended in oil, to be performed below 200°. Burning of the oil vehicle is thus avoided.

T. S. WHEELER.

Treatment of oils and fats to neutralise and remove their fatty acid content. J. W. SPENSLEY (U.S.P. 1,648,367, 8.11.27. Appl., 5.3.23. U.K., 5.5.22).—See B.P. 201,968; B., 1923, 962 A.

Preparatory treatment of cottonseed [for extraction of oil]. H. BOLLMANN, Assr. to M. F. FOSTER (U.S.P. 1,653,201, 20.12.27. Appl., 17.8.23. Ger., 29.8.22).—See G.P. 393,072; B., 1924, 603.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Water-resistance tests [on paints and varnishes].

H. WOLFF (Farben-Ztg., 1928, 33, 921—923).—In a series of experiments on water-resistance of paints and varnishes under varying conditions, conflicting results were obtained according to whether the immersion was continuous or intermittent, and whether temporarily or permanently hard water or distilled water was used. The following standard method of procedure is suggested:—Films (one or two coats, according to the type of material) on clean glass and iron plate are half-immersed in water after 48 hrs. air drying. If tap water be used, it should be freed from temporary hardness. The immersion is continuous, daily examination being made for milkiness, softening, etc. A parallel series of tests should be run, the films being allowed to dry out after each 2 days' immersion, and the reversibility of any changes observed. S. S. WOOLF.

Ageing of boiled oil. F. WILBORN (Farben-Ztg., 1927, 33, 864).—Over a period of 10 months, samples of boiled oil were prepared monthly by incorporating a standard amount of lead-manganese resinate (the same "melt" being used throughout) in the same well-preserved linseed oil. A similar series was prepared using cobalt linoleate as drier. At the end of this time no appreciable sedimentation had occurred in any sample, and the drying times of the oils showed no significant "ageing" variation. This is contrary to previous statements to the effect that the driers in boiled oil gradually lose effectiveness by precipitation. S. S. WOOLF.

Influence of driers on the drying of oil of amber.

F. WILBORN (Farben-Ztg., 1927, 33, 862—864).—Additions of lead-manganese resinate and of cobalt resinate to redistilled oil of amber are graphically shown to accelerate the drying by catalysing resinification. The results are briefly discussed. S. S. WOOLF.

Physical chemistry of colour lake formation.

III. Alizarin lakes. H. B. WEISER and E. E. PORTER (J. Physical Chem., 1927, 31, 1824—1839).—The mechanism of the formation of alizarin lakes has been studied by measuring the adsorption of alizarin and alizarin SW by sols of the hydroxides of iron, chromium, and aluminium at varying p_H values. Lake formation is due to adsorption of the dye anion by the hydrous oxides in amounts depending on the composition of the dye bath. The adsorption curves for alizarin SW and the aluminium and chromium sols show no evidence of the formation of a compound between the dye and the mordant at any p_H value, the influence of which is similar to that on sulphate and oxalate (A., 1927, 1021), the decreasing order of adsorption from neutral and basic baths being alizarin SW, oxalate, and sulphate. Experiments on the adsorption from sodium alizarate baths show the dye anion to be adsorbed, leaving most of the sodium in the bath, thus disproving the suggestions of Bull and Adams (A., 1922, i, 355), Williamson (A., 1924, i, 1080), and Biltz (A., 1906, ii, 78) as to the nature of lake formation. The effects of the sulphate ion, the calcium ion, and of calcium sulphate at varying p_H values have also been studied. The sulphate ion replaces the dye if the bath is acid, but not if alkaline; the

calcium ion increases the charge on the mordant, thus permitting greater adsorption of the dye anion, and the data with calcium sulphate show that the effects of the individual ions are independent of each other, each, in turn, being dependent on the hydrogen-ion concentration.

L. S. THEOBALD.

British standard specifications for pigments.

C. A. KLEIN (J. Oil and Colour Chem. Assoc., 1927, 10, 355—372).—A general account of the inception of the British Engineering Standards Association Subcommittee on paint materials, with special reference to the work of the Pigment Panel. The nature of the specifications evolved for dry pigments and oil-pastes is indicated, the various clauses being briefly discussed.

S. S. WOOLF.

Nitro-oil- or combination-lacquers.

F. KOLKE (Farben-Ztg., 1927, 33, 861—862).—The combination of nitrocellulose varnishes with oil varnishes is discussed. Curves are given showing the limits of compatibility of tung oil with 20% $\frac{1}{2}$ -sec. nitrocellulose solutions in butyl acetate and in hexalin acetate. S. S. WOOLF.

PATENTS.

Manufacture of composite titanium pigments.

H. WADE. From TITANIUM PIGMENT Co., INC. (B.P. 281,459, 9.11.26).—A composite titanium oxide-calcium sulphate pigment is prepared by heating a solution of a titanium compound, *e.g.*, titanium sulphate, in which is suspended calcium sulphate previously prepared in acicular crystalline form (either by precipitation in alkaline or acid media or by calcination of natural or by-product calcium sulphate). The composite precipitate is neutralised, washed, and calcined in the presence of calcium phosphate or other agent inhibitive of deteriorative colour change. S. S. WOOLF.

Manufacture of [phenol-acetaldehyde] condensation products. H. W. MATHESON, ASSR. to CANADIAN ELECTRO PRODUCTS Co., LTD. (U.S.P. 1,653,302, 20.12.27. Appl., 19.8.22).—Phenol and acetaldehyde are condensed at 50—80° under pressure in the presence of less than 1% of a condensing agent comprising a halogen derivative of a non-metal. S. S. WOOLF.

Production of phenol-methylene resins and alcohols from methylals. C. B. CARTER, ASSR. to S. KARPEN & BROS. (U.S.P. 1,645,226, 11.10.27. Appl., 26.8.25).—A phenol is boiled at atmospheric pressure for a few minutes with a methylal in presence of concentrated sulphuric acid (10—20%) to give a hard, fusible, brittle resin and the alcohol corresponding to the methylal employed. T. S. WHEELER.

Manufacture of synthetic resins. B. N. LOUGOVY, ASSR. to ELLIS-FOSTER Co. (U.S.P. 1,648,852, 8.11.27. Appl., 23.10.24).—Benzotrichloride (1 mol.) is treated with a phenol, *e.g.*, *o*-cresol (3 mols.); the product (containing a dye) on being heated with formaldehyde solution yields a resin which can be moulded hot and hardened with hexamethylenetetramine.

T. S. WHEELER.

Manufacture of plastic compositions. E. C. R. MARKS. From SELDEN Co. (B.P. 281,742, 7.7.26).—Ethyl phthalate or a similar normally liquid, non-resinifying ester of b.p. 250° or over is incorporated with a resinous substance capable of being hardened

by heat, *e.g.*, phenol-aldehyde condensation products, glyceryl phthalate, shellac, etc. The ester is added to the resinous material (in the fusible state) in amount precluding substantial separation of the components during the subsequent hardening process.

S. S. WOOLF.

Treatment of resins for making varnish, paints, etc. J. F. BENNETT and J. HADFIELD (B.P. 281,377, 31.8.26).—A varnish base is prepared by treating a resin, alone or admixed with linseed or other oil, with nitric acid, preferably of *d* 1.3. Heat may be used to start the reaction, but the temperature must be kept below 100°. After neutralisation, the product is dissolved in suitable solvents and mixed with nitrocellulose, oils, resins, bitumens, etc., pigments being incorporated if desired.

S. S. WOOLF.

Decolorising, treating, and dissolving shellac for bleaching purposes. F. C. RAWOLLE (U.S.P. 1,644,191, 4.10.27. Appl., 31.8.23).—Crude shellac after being shredded in hot water is treated with sodium carbonate solution in a vessel fitted with means for further subdividing the material to promote dissolution.

T. S. WHEELER.

[Manufacture of writing] ink. H. SCHLADERBACH and H. HÄHLE, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,645,117, 11.10.27. Appl., 8.1.26. Ger., 3.1.25).—See B.P. 267,633; B., 1927, 387.

[Manufacture of colour] lakes. E. HARTMANN, ASSR. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,653,851, 27.12.27. Appl., 27.12.23. Ger., 22.5.23).—See B.P. 216,486; B., 1925, 78.

Manufacture of synthetic resin. A. BLUMFELDT and H. KAEGI, ASSRS. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,654,856, 3.1.28. Appl., 19.3.24. Switz., 9.4.23).—See G.P. 401,168; B., 1925, 216.

Manufacture of [urea-acr]aldehyde condensation product. A. GAMS and G. WIDMER, ASSRS. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,654,215, 27.12.27. Appl., 7.10.26. Switz., 22.10.25).—See B.P. 260,288; B., 1927, 419.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Influence of emarex (M.R.X.) on the physical properties of vulcanised rubber. L. STOLL (Gummi-Ztg., 1927, 42, 695—696).—When added to a mixture of rubber (100 pts.), zinc oxide (5 pts.), sulphur (5 pts.), and diphenylguanidine (1½ pts.) the above form of "mineral rubber" (up to 5% on the rubber) causes a distinct acceleration of vulcanisation and an increased extension of the product at 200 kg./sq. cm. With higher proportions the extension becomes less and then again increases without attaining the value for the standard mixing. The extension at break reaches a maximum at 30%. Permanent set increases and hardness decreases with rising percentages of the mineral rubber.

D. F. TWISS.

Transformation temperature for rubber at approximately 60—80°. L. STOLL (Gummi-Ztg., 1927, 42, 745—746).—As evidence of the existence of a transformation of rubber between 60° and 80°, instances are cited of the importance of this temperature range for

various processes and changes with latex and with raw vulcanised rubber.

D. F. TWISS.

PATENTS.

Manufacture of hydrocyclocaoutchouc. H. STAUDINGER (B.P. 263,862, 30.12.26. Switz., 31.12.25).—*cycloCaoutchouc*, obtained by the action of heat on rubber, can be hydrogenated, preferably above 200° and under an increased pressure of hydrogen, in the presence of a catalyst, desirably a metal of the 8th series of the periodic system. A solvent may be used to keep the *cyclocaoutchouc* in solution during the reduction or to remove the resulting *hydrocyclocaoutchouc* subsequently from the catalyst. *Hydrocyclocaoutchouc* has n_D^{20} 1.5263 and is useful for the manufacture of plastic masses and as a substitute for gutta-percha.

D. F. TWISS.

Attaching rubber to metal, glass, wood, and similar rigid surfaces. R. M. WITHEYCOMBE (B.P. 256,227, 26.7.26. U.S., 29.7.25).—Rigid surfaces, coated with a vulcanisable rubber solution or paint and dried, are subjected to a dry heat ranging from 105° to 260° at atmospheric pressure. The resulting coating may vary from a thin, hard, enamel-like layer to one of highly polished vulcanite. [Stat. ref. to B.P. 8757 of 1913.]

D. F. TWISS.

Method of deodorising [putrefaction products of rubber latex]. S. M. CADWELL, ASSR. to NAUGATUCK CHEMICAL Co. (U.S.P. 1,648,259, 8.11.27. Appl., 18.2.26).—The putrefaction products of nitrogenous materials, *e.g.*, rubber latex, are deodorised by addition of a non-nitrogenous organic oxide, sulphide, or disulphide containing more than one carbon atom in the molecule, and also a double linking adjacent to a carbon atom, *e.g.*, benzoyl peroxide, acetic anhydride.

T. S. WHEELER.

Treatment [neutralisation] of rubber latex [containing ammonia]. H. W. KELLEY and W. D. WOLFE, ASSRS. to UNITED SHOE MACHINERY CORP. (U.S.P. 1,644,730, 11.10.27. Appl., 16.3.25).—A mixture of boric acid and glycerol is employed.

T. S. WHEELER.

Manufacture of products resembling rubber. CONSORTIUM F. ELEKTROCHEM. IND., G.M.B.H. (B.P. 261,748, 16.11.26. Ger., 23.11.25).—Polymerised vinyl compounds, *e.g.*, polymerised vinyl alcohol or a homologue or derivative such as a polymerised vinyl ester, ether, or acetal, are treated with a vulcanising agent such as sulphur (aided by heat and, if desired, an accelerator) or sulphur chloride. According to the nature of the original material and the conditions of treatment the products may be obtained with the characteristics of soft rubber or vulcanite and with other specific properties.

D. F. TWISS.

Treatment of rubber articles. GUTTA PERCHA & RUBBER, LTD., Assees. of J. J. MORIARTY (Can. P. 264,816, 11.12.25).—The sulphur content of the surface layer of articles constructed of soft rubber is increased by treating the articles with sulphur chloride solution.

L. A. COLES.

Manufacture of a caoutchouc derivative. H. STAUDINGER, ASSR. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,654,844, 3.1.28. Appl., 22.12.26. Switz., 31.12.25).—See B.P. 263,862; preceding.

Vulcanisation of caoutchouc. S. J. PEACHEY and A. SKIPSEY (U.S.P. 1,653,821, 27.12.27. Appl., 19.3.25. U.K., 26.3.24).—See B.P. 242,464; B., 1926, 68.

Vulcanising of caoutchouc. C. W. BEDFORD, ASSR. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,645,084, 11.10.27. Appl., 11.7.23).—See B.P. 130,857; B., 1919, 731 A.

Treatment of latex and products thereof. NAUGATUCK CHEMICAL CO., ASSEES. of J. MCGAVACK (B.P. 268,299, 31.1.27. U.S., 24.3.26).—See U.S.P. 1,647,805; B., 1928, 62.

XV.—LEATHER; GLUE.

Synthetic tannins. L. MEUNIER and C. GASTELLU (Bull. Soc. d'Encour., 1927, 126, 732—742).—Equal weights of phenol and sulphuric acid of 104.5% strength should be used in sulphonating phenol. The phenol is melted at 40° and poured in a thin stream into the sulphuric acid which is stirred meanwhile. The temperature rises quickly to 150°; the mixture is allowed to cool to 100°, at which temperature it is kept for 2½ hrs. Under these conditions, 200 pts. of phenol yield 400 pts. of a product which melts at 25—30° to a brown liquid, is entirely soluble in water, and contains 351 pts. of phenol-sulphonic acid, 4.8 pts. of free sulphuric acid, and 11 pts. of free phenol. When the crude product is condensed with formaldehyde, the phenol-sulphonic acid itself forms a soluble compound, whereas the free phenol condenses with formaldehyde to form insoluble resins which are partially peptised by the excess sulphuric acid and the other condensation products. In practice, crude phenol-sulphonic acids containing less than 5% of free phenol are condensed with formaldehyde to form products entirely soluble in water. The process is readily completed if 1 mol. of formaldehyde is used for every 2 mols. of phenol-sulphonic acid. The consistency of the product and its solubility in water diminish proportionately with the increase in the amount of formaldehyde used. These variations in the properties result from the increased degree of polymerisation. The condensation products should be neutralised until the total residual acidity corresponds to 14—15 g. of sulphuric acid per 100 g. of phenol originally used. With such a product, the fixation of the tannin by the skin takes place rapidly and attains a maximum of 14—15 pts. of tannin per 100 pts. of leather. D. WOODROFFE.

Tanning materials of the British Empire. II. (Bull. Imp. Inst., 1927, 25, 380—403).—A review of oak and larch barks, chestnut and cutch woods, and gambier and sumach.

Tanning, currying, and dyeing of [dog] skins. R. MACH (Gerber, 1927, 53, 61—62, 71—73; Cliem. Zentr., 1927, II, 659).—The hides, which are shorn mechanically and the long hairs removed, are softened and fleshed, and a bate containing salt, alum, acids, and barley groats is brushed several times on to the skin. After drying, the skins are greased and pared, and then cleaned by treatment in heated rotating vessels with sawdust, white sand, and alabaster. The hair must be treated with salts and acids before dyeing. Ursol and Nako dyes are developed on the hair with oxidising agents, yielding brown, grey, or black shades; if a deep

black is required, dyewoods, Aniline Black, or vat dyes are used. L. A. COLES.

Gelatin. J. S. ALLAN (Proc. Tech. Sect. Paper-makers' Assoc., 1926, 7, 69—87).—The properties of gelatin, more especially in relationship to the sizing and coating of paper, are reviewed. D. J. NORMAN.

PATENTS.

Preparation of skins of fish, such as and in particular those of sharks, for tanning. A. EHRENREICH (B.P. 281,918, 24.3.27. Belg., 25.1.27).—The skins are limed for a considerably reduced period, then treated with enzyme products obtained from an intestinal gland of the fish or pancreatic extracts, respectively, alone or with the fluid gall or the urine of the fish.

D. WOODROFFE.

Preparatory treatment of skins of fish, such as, and in particular, the plagiostomi. A. EHRENREICH (B.P. 281,919, 24.3.27. Belg., 26.1.27).—The skins are given a very short liming, treated with enzymes, then agitated in a pit or drum containing about 5% of gum tragacanth, extract of algae, or extract of fenugreek for about 6 hrs. at approximately 30°. The skins are afterwards tanned, and may be waterproofed by impregnating them after drying with a benzene solution of rubber or calcium stearate.

D. WOODROFFE.

Tanning of white leather. H. MORIN (F.P. 615,126, 10.9.25).—The pelts are drummed for several hours in a solution containing 15 pts. of sodium hydrogen sulphate and 15 pts. of sodium chloride in 160 pts. of either cold or warm water. They are then drained and air- or machine-dried at a temperature not above 50°. After neutralising the dry hides with alkali (preferably sodium silicate) together with a little sodium chloride to facilitate the penetration of the alkali, the hides are piled in slightly moist sawdust. The product is a white soft leather, and the tannage is economical.

D. WOODROFFE.

Neutralising leather after tannage or bleaching. L. JABLONSKI (G.P. 442,233, 5.9.22).—Vegetable-tanned leather is bleached by dipping it for a few seconds in a very dilute solution of sodium carbonate, and then plunging it into a very dilute solution of sulphuric or oxalic acid. It is then neutralised by immersion for 30 sec. or longer in a weak solution (2%) of aniline acetate, hydrazine compounds, or an organic base or its salts. Alum-tanned leather which has hitherto not been neutralised can be rendered more water-resistant by treatment with such compounds, e.g., aniline or pyridine salts.

D. WOODROFFE.

Manufacture of chamois leather. A. J. CLERMONT (Addn. No. 31,603, 17.11.25, to F.P. 595,954; B., 1927, 758).—The sodium carbonate solution of the prior patent can be replaced by aqueous solutions of sodium, potassium, or magnesium hydroxides, potassium carbonate, or the sulphates, nitrates, chlorides, neutral or acid sulphites of the alkali metals and magnesium, as well as by sodium or potassium hypochlorite. After the hides or pelts have been treated on the flesh side with one of these solutions, and washed in water, they are treated with linseed oil, glucose, or the soluble commercial oils and finished.

D. WOODROFFE.

Impregnating leather and pelts and rendering them elastic. L. J. DOUMEYROU (F.P. 609,296, 16.4.25).—Ammonium salts of the higher fatty acids are prepared by treating the fatty acids or fats with gaseous ammonia or with ammonium salts, *e.g.*, ammonium carbonate, which readily decompose. These salts are dissolved in benzene, benzene and alcohol, petroleum, or alcohol and ether and treated with water to form an emulsion, solution, or paste which is worked into the leather. The latter is washed and dried.

D. WOODROFFE.

Weighting of leather and production of a glossy surface thereon. ASSOC. PARISIENNE POUR L'IND. CHIM., Assecs. of E. DESPARMET and F. SCHMITT (F.P. 615,952, 1.10.25).—The raw leather is treated with metallic salts (*e.g.*, aluminium, zinc, manganese, bismuth) of the higher saturated or unsaturated fatty acids (*e.g.*, caproic, caprylic, capric, lauric, palmitic, erucic acids, etc.), or mixtures of these with each other or with other substances. It is then ironed to produce a glossy surface. The process hides defects better and renders the leather firmer than does talc, bismuth salt, or zinc oxide, and varnishes or cellulose lacquers give a better finish.

D. WOODROFFE.

Drying of leather, hides, skins, etc. V. G. WALSH (B.P. 280,770, 7.3.27).—The epithelial surface of the tanned leather is treated with a solution of a protein such as gelatin to precipitate the surface tan, after which it may be oiled with the usual oils and the flesh side covered to a depth of 0.25 in. or more with a layer of anhydrous sodium sulphate alone or mixed with sand. The leather so treated is kept for 24–30 hrs., after which the layer is removed and the leather finished as usual. Untanned skins, hides, etc. may be dried out by applying a suitable drying agent to the flesh side.

D. WOODROFFE.

Treating [waterproofing] leather. T. BLACK-ADDER (U.S.P. 1,645,642, 18.10.27. Appl., 1.10.25).—An emulsion of a mineral oil in an aqueous sodium soap solution is reversed by treatment with aluminium sulphate solution, and the resulting water-in-oil emulsion containing an aluminium soap is employed to impregnate leather.

T. S. WHEELER.

Manufacture of artificial horn. J. ELLENBERGER, ASSR. to BRIT. GLUES & CHEMICALS LTD. (U.S.P. 1,649,675, 15.11.27. Appl., 12.4.26. Ger., 9.1.25).—See B.P. 271,221; B., 1927, 565.

XVI.—AGRICULTURE.

Influence of calcium carbonate on the soil. A. A. RODE (Izvestia Leningrad Lesnogo Inst., 1927, 34, 119–180).—Experiments with podsol and artificially unsaturated chernozem soils show that on the addition of calcium carbonate the excess of acid is neutralised with disturbance of the chemical and biological equilibria, and that organic substances are thereby decomposed; the decomposition ceases when a new equilibrium is reached whereby the calcium ions decrease the dispersion.

CHEMICAL ABSTRACTS.

Effects of calcium compounds on the soil and on plant growth. W. T. H. WILLIAMSON (Scottish J.

Agric., 1927, 10, 180–184).—Mineral phosphate reduced the acidity and increased the exchangeable calcium content of soil, but not so effectively as calcium carbonate. Heavy applications of superphosphate or calcium chloride caused a temporary (6–12 months) increase in soil acidity (that from calcium chloride destroying all vegetation) and a considerable increase in exchangeable calcium.

CHEMICAL ABSTRACTS.

Effect of frost on soils. H. GORKA (Koll.-Chem. Beih., 1927, 25, 127–175).—On exposure to frost, clay suspensions undergo an increase in viscosity. This and the similar effect due to mechanical treatment are considered to be due to a disintegration of aggregated particles, with liberation of matter still capable of swelling. In agreement with the view expressed above, it was observed in sedimentation experiments that the total volume of material deposited was greater from a suspension which had been frozen than from an unfrozen suspension; also the rate of settling of the particles was greater in the suspension which had been frozen. Experiments on the velocity of cataphoresis of the particles showed that mechanical agitation of the suspension increases the velocity of migration, probably as a result of the liberation of electrolytes from inside the aggregates. Frost has the effect of reducing the velocity of cataphoresis, but this is raised to a value above the original by subsequent mechanical treatment.

E. S. HEDGES.

Influence of sulphur and soluble sulphur compounds in soil on nitrification. G. GUITTONNEAU (Compt. rend., 1927, 185, 1518–1520).—Addition of sulphur to soil containing peptone and calcium carbonate causes an arrest in the nitrification up to about 60 days, after which nitrification begins and the intermediate sulphur compounds disappear. The effect of organisms on ammonium sulphate, ammonium sulphate + sulphur, and ammonium thiosulphate, in silica gels, has been studied, and it is found that sulphur does not stop nitrification when it is transformed directly into sulphate. Ammonium thiosulphate causes a hindrance in the microbic oxidation of ammonia.

H. BURTON.

Effect of liquid manure on soils and utilisation of the potassium and phosphoric acid of liquid manure by plants. J. C. FICK (J. Landw., 1927, 75, 215–256).—It is concluded from laboratory experiments with liquid manure and with the urine of farm animals that neither has a solvent action on soil nutrients, and that the nutrients present are absorbed by the soil. The physical condition of the soil is impaired by application of urine or liquid manure, hygroscopicity being increased and permeability diminished. The results of vegetation experiments with maize indicate that the phosphoric acid and potassium in liquid manure are less efficient than equivalent amounts supplied as mineral manures, owing probably to the harmful effect of the material on the physical condition of the soil.

C. T. GIMMINGHAM.

Manuring of heavy soils rich in potash. DIENST (Ernährung der Pflanze, 1927, No. 6; Bied. Zentr., 1927, 56, 549–551).—Two stiff clay soils, shown by the Neubauer method to contain ample available potash,

nevertheless proved very responsive to potash fertilisers when cropped with roots and potatoes.

A. G. POLLARD.

[Manurial] investigations at the Königsmoor experimental station. B. TACKE (*Landw. Jahrb.*, 1927, **65**, 8—43; *Bied. Zentr.*, 1927, **56**, 545—549).—Manurial experiments on moorland meadows indicated the superiority of 40% potash salts over kainit. The amount of lime needed was unaffected by the nature of potash manures used. Excessive liming decreased hay yields. The hay crop was not noticeably improved by nitrogenous fertilisers or compost, and drainage reduced the crop. Chalking the subsoil improved the crop. On permanent pasture similar results were obtained. Periodical moving of the grazing cattle and intermittent mowing were without effect on the meat-producing power of the herbage. On cropped moorland soils sodium nitrate was a more effective fertiliser than ammonium sulphate, as the latter markedly lowered the lime reserve. Heavy liming reduced the yield of both grain and straw of oats, but increased the yields of potatoes.

A. G. POLLARD.

Influence of ammonium sulphate as a direct source of nitrogen for apple trees. M. B. DAVIS (*Sci. Agric.*, 1927, **8**, 41—55).—The nitrogen of ammonium sulphate becomes available only when nitrifying organisms are present. When these are absent, sodium nitrate is less toxic than cyanamide or ammonium sulphate.

CHEMICAL ABSTRACTS.

Nitrate utilisation by plants. I. Régime of nitrate nitrogen under natural conditions of plant development. A. SHMUK (*Ann. Kuban Agric. Inst. [Russia]*, 1925, **3**, 77—94).—Soils entirely covered with vegetation contain little or no free nitrates. The root-mass stimulates the activities of denitrifying organisms and the denitrifying enzymes of the plants. In solution, nitrates are reduced by roots to nitrites and ammonia; ammonia should therefore be a source of nitrogen for plants.

CHEMICAL ABSTRACTS.

Relation of the yield and protein content of wheat to the nitrogen content of the soil. R. E. NEIDIG and R. S. SNYDER (*Idaho Res. Sta. Bull.*, 1926, **5**, 3—32).—Under the prevailing conditions, the protein content of wheat was increased by manuring and by summer fallowing; the latter reduces the organic matter, increases the availability of plant foods (particularly nitrogen), and causes greater erosion. Manuring causes a better utilisation of water. The total plant foods are more rapidly depleted, crop for crop, by fallowing one year in three than by cropping every year with suitable rotation. The protein content of the wheat was 12.03—15.05%.

CHEMICAL ABSTRACTS.

Physiological reaction of "nitrophoska." R. W. BELING (*Z. Pflanz. Düng.*, 1927, **6B**, 562—567).—As a nutrient in water-culture experiments "nitrophoska" produces no change in the reaction of the nutrient. In soils, however, it shows definite physiological acidity, increasing both hydrolytic and exchange acidity and reducing the buffer capacity. This effect is considerably less than that of ammonium sulphate on an equivalent nitrogen basis.

A. G. POLLARD.

Utilisation of phosphorite phosphoric acid by plants. V. JOHANSON (*Bot. Arch.*, 1927, **14**, 319—454; *Chem. Zentr.*, 1927, **i**, 2558).—Physiologically acid fertilisers lead to poorer plant growth than physiologically neutral ones, in spite of a greater absorption of phosphorus. The effect of calcium and iron was examined.

A. A. ELDRIDGE.

Superphosphate as stimulant of [plant] growth. L. ZALESKI (*Mem. Inst. Nat. Pol. Econ. rurale Pulawy*, 1926, **7**, 27 pp.; *Chem. Zentr.*, 1927, **i**, 2598).—A study of the effect of the introduction of small quantities of superphosphate immediately under the seeds.

A. A. ELDRIDGE.

Three-year comparison of Neubauer analyses and field trials. T. ROEMER, DIRKS, and WOACK (*Z. Pflanz. Düng.*, 1927, **6B**, 529—562).—The Neubauer limiting values for root-soluble phosphate varied with the nature of the crop. Average values obtained were: rye and oats 4 mg.; potatoes 4—5 mg.; grass land 6 mg.; barley (varies considerably with season) 6 mg.; sugar beet 6—7 mg. There was a general agreement between Neubauer results and field trials, but marked divergences were apparent in some crops. In potash trials agreement was not quite so good, best results being obtained with potatoes. Neubauer values obtained for limiting values for potash were:—Oats (variable) and rye 20 mg.; grass 25 mg.; barley 25—30 mg.; wheat (very irregular) 30 mg.; potatoes 36—42 mg.; and sugar beet 25 mg. Neubauer results for available nutrients when differing from those of field trials were more often high than low. Differences are partially explicable when the different experimental conditions are considered. In the Neubauer method the effect of the subsoil nutrients is not considered, and, further, the preparation of the soil sample favours a temporarily increased bacterial activity resulting in higher nitrate contents than obtain in corresponding field soils. Temperature and moisture changes during the experimental period may differ considerably in the two cases. The p_H values of soils may be altered by the Neubauer treatment. This effect is not marked in nearly neutral soils, but more extreme values tend to be intensified by the experimental conditions. The Neubauer method is considered a valuable basis for the determination of fertiliser requirements, but needs further development.

A. G. POLLARD.

Temperature effects in the metabolism of wheat. W. E. TOTTINGHAM [with E. J. RANKIN, A. D. DICKSON, and H. W. LOUWSME] (*Plant Physiol.*, 1926, **1**, 307—336).—Atmospheric humidity has little influence on the protein content of wheat during seed development. In fully-nourished plants, illuminated by 530—1150 ft.-candles over a 12 hr. day, the production of dry matter was favoured by the lower temperature plane, and the percentage of protein was increased at the expense of the available carbohydrates. With 1560—2510 ft.-candles per 12 hr. day, plants harvested at one third and one fourth of full growth contained more sucrose and protein at lower temperatures; mature plants contained increased percentages of carbohydrates, and gave much greater yields, especially of grain, at the lower temperatures.

CHEMICAL ABSTRACTS.

Climatic effects in the metabolism of maize. W. E. TOTTINGHAM and H. W. KERR (*Plant Physiol.*, 1926, 1, 415—416).—The percentage of sucrose in the dry matter of the leaf greatly exceeded that of reducing sugars. Both sucrose and dextrin varied with solar radiation. The sugars and monoamino-acids in the stalk and ear varied independently of their proportions in the leaf. The stability of maize and sorghum compared with wheat towards climatic influences may be due to their higher content of sucrose and less soluble forms of protein and lower content of dextrose, soluble proteins, and other soluble nitrogenous compounds.

CHEMICAL ABSTRACTS.

Seasonal variations in the carbohydrate content of swedes. J. CALDWELL (*Scottish J. Agric.*, 1927, 10, 325—332).—The total carbohydrate content of Scotia swedes was highest in roots harvested early in December, but the highest percentages of dry matter and carbohydrate were present in roots harvested at the end of September.

CHEMICAL ABSTRACTS.

Determination of hardness in apple varieties and the relation of some factors to cold resistance. A. C. HILDRETH (*Minn. Agric. Exp. Sta. Tech. Bull.*, 1926, No. 42).—High reserves of carbohydrates and organic nitrogen are possibly correlated with greater hardness.

CHEMICAL ABSTRACTS.

Value of the chemical test in the identification of wild white clover. W. M. FINDLAY and G. DOWER (*Scottish J. Agric.*, 1927, 10, 219—224).—When crushed seedlings of wild white clover are incubated for several hours at 32°, hydrogen cyanide (detected with sodium carbonate-picric acid test papers) is produced. Large and small seeds of pure wild white clover give equally intense reactions.

CHEMICAL ABSTRACTS.

Petroleum oil as an insecticide for citrus trees. E. R. DE ONG, H. KNIGHT, and J. C. CHAMBERLAIN (*Hilgardia*, 1927, 2, 351—384).—A preliminary study. Injury is associated with a high percentage of unsaturated hydrocarbons.

CHEMICAL ABSTRACTS.

Determination of chlorophyll by Willstätter's method. T. STECHE (*J. Landw.*, 1927, 75, 211—214).—As the result of a large number of determinations of chlorophyll in potato plants and grasses by the method of Willstätter and Stoll, some changes in the direction of simplifying and shortening the process are proposed. The plant material is treated directly with pure acetone, preliminary treatment with aqueous acetone being omitted, since the yellow-brown pigment is so little soluble in ether that it is completely extracted by the water used for washing out the acetone after addition of ether. Difficulties were met with in certain cases in the washing of the ether solution owing to the presence of colloidal material. The use of 100 c.c. of 1% potassium chloride solution for the first five washings and 100 c.c. of 3% solution for the sixth, instead of water only, flocculated the colloids and gave better separation. It was further found advantageous to carry out saponification of the chlorophyll in three stages, using 5 c.c. of a saturated solution of potassium hydroxide in methyl alcohol at each stage.

C. T. GIMINGHAM.

Determination of total carbon and carbon dioxide in small quantities of soil solutions. C. F. FLINT

(*J.C.S.*, 1927, 2975).—A wet combustion method used in steel analysis is adapted to the routine examination of soil solutions by employing in the combustion-furnace tube a cylinder of reduced copper gauze together with either copper oxide and a spiral of silver gauze or a cartridge of lead chromate. Oxidation of the test solution is carried out with a boiled-out mixture of chromic and sulphuric acids, all volatile organic matter and other volatile substances being disposed of by the reagents in the combustion furnace tube. The carbon dioxide is absorbed and weighed in soda-lime. Consistent results were given on repeated tests on soil solutions. In determining only carbon dioxide the furnace tube is removed. Dilute hydrochloric acid is used for the liberation of carbon dioxide from the test solution, its expulsion being assisted by the passage of a current of purified air and the simultaneous generation of hydrogen in the reaction flask.

R. A. PRATT.

Dissolution and peptisation of humic acid by sodium hydroxide. Wo. OSTWALD and W. RÖDIGER (*Kolloid-Z.*, 1927, 43, 225—227).—Experiments were made to determine the solubility of humic acid in sodium hydroxide solutions of various concentrations and in presence of varying amounts of the solid phase, with the object of ascertaining whether the process is purely chemical or is due to peptisation. When the concentration of alkali is extremely small (0.0005*N* to 0.001*N*), and again at a higher concentration (0.025*N*), the curves connecting the amount of dissolved humic acid with the amount of solid phase show that the dissolution can be ascribed solely to the production of alkali humate. At intermediate concentrations the curves pass through a maximum, showing that peptisation occurs in addition to the chemical reaction. In working on the chemical reactions of humic acid it is recommended to use a large excess of the solid phase, so eliminating the effect of peptisation.

E. S. HEDGES.

Storage of beets. DOCHLENKO.—See XVII.

PATENTS.

Treatment of oat seed to destroy smut spores. I. G. FARBENIND. A.-G. (B.P. 275,539, 15.10.26. Ger., 5.8.26).—Treatment of oat seed with a dry powder consisting of a quinone, or quinone derivative, or an additive compound of a quinone and a phenol (*e.g.*, *p*-chlorophenoquinone), mixed with a diluent, renders it substantially free from living spores of smut fungi. The germinating capacity of the seed is unaffected.

C. T. GIMINGHAM.

Cultivation of edible fungi [mushrooms]. J. SZÜCS (B.P. 270,684, 27.4.27. Austr., 6.5.26).—A solid or semi-solid carrier, *e.g.*, peat, straw, sawdust, earth, dry leaves, pumice stone, agar-agar, etc., is mixed with nutrient materials such as molasses and salts containing phosphorus and nitrogen, and infected with cultures of suitable bacteria or mould fungi in order to produce a fermentation similar to that of horse-manure. The resulting nutrient medium, after sterilising if desired, preferably by heat, is spawned in the usual way by direct spore-culture methods (with spores or preliminarily germinated spores) or with mycelium not directly

derived from spores, when the fungi develop uniformly at room temperature. F. R. ENNOS.

Manufacture of insecticides. [A] H. P. CORSON and [B, c] W. K. SCHWEITZER, Assrs. to GRASSELLI CHEMICAL Co. (U.S.P. 1,648,577 and 1,648,595—6, 8.11.27. Appl., 29.9.24).—(A) The process of U.S.P. 1,591,795 (B., 1926, 743) is modified in that the product is heated with water and calcium hydroxide for 24 hrs. (B) The processes of (A) and of the patent cited are combined in one operation. (c) The process described in (A) is modified in that manganese carbonate is added with the calcium hydroxide. T. S. WHEELER.

Insecticide emulsion. P. R. JONES, Assr. to BALFOUR, GUTHRIE, & Co. (U.S.P. 1,646,149, 18.10.27. Appl., 20.8.25).—An emulsion of kerosene in a mixture containing water, calcium caseinate, dextrin, and bentonite, and, preferably, lignin liquor and maize oil soap is of value as a spray for fruit trees. T. S. WHEELER.

Insecticides (B.P. 280,256).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Sugar losses in the storage of beets. J. J. DOCHLENKO (Zapiski, 1926—7, 4, 77—86).—The rolled portions of beets contain no sucrose, but much invert sugar and high acidity; mouldy beets contained less sucrose, more invert sugar, and higher acidity than sound beets. Treatment with lime milk (*d* 1.05) was most effective. On long storage the harmful nitrogen increased, the total- and albumin-nitrogen decreased, and the amino- and ammonia-nitrogen remained unchanged.

CHEMICAL ABSTRACTS.

Desaccharification of molasses by the acetic acid method. G. MEZZADROLI, I. MUTTI, and A. PIOMBO (Zymologica, 1927, 2, 121—136).—The experimental results obtained confirm the possibility of desaccharifying molasses by the acetic acid process (cf. Rajtora, B., 1925, 254). The industrial feasibility of the process depends on the prices of sugar, molasses, and acetic acid, the extent of the loss of acetic acid, and the cost of redistilling and concentrating the recoverable acid.

T. H. POPE.

Testing of potato starch. O. WOLFF (Chem.-Ztg., 1927, 51, 1001—1003).—The suitability of potato starch for the preparation of adhesive paste is determined by means of two tests. The starch is ground with eleven times its weight of distilled water and the mixture kept for 24 hrs., after which time it is tested by impact with the hand. For the second test, a paste is prepared under standard conditions, using absolutely pure water, and the viscosity determined by the falling-ball method. A suitable starch is used as standard, and a numerical expression for the degree of suitability of the sample is obtained.

S. I. LEVY.

PATENTS.

Production of crystallised sugar from molasses and syrup. Soc. ANON. DES DISTILLERIES DES DEUX-SÈVRES (F.P. 621,075, 3.9.26. Belg., 5.9.25).—Anhydrous ethyl acetate, or another alkyl acetate, is added slowly with stirring to a mixture of molasses or syrup with acetic acid, whereby the sucrose is rendered insoluble and inversion is prevented.

L. A. COLES.

Oxidation of aldoses. A. STOLL and W. KUSSMAUL, Assrs. to CHEMICAL WORKS (FORMERLY SANDOZ) (U.S.P. 1,648,368, 8.11.27. Appl., 16.5.27. Ger., 12.6.25).—Sodium hypochlorite can be used in place of sodium hypobromite or hypoiodite for the oxidation of aldoses to the corresponding monocarboxylic acids if a small quantity of sodium bromide or iodide be added to the reaction mixture. T. S. WHEELER.

Preparation of sugar cane for extracting juice from cane stalks. W. H. MORGAN, SEN., Assr. to W. H. MORGAN, JUN. (U.S.P. 1,655,327, 3.1.28. Appl., 19.8.25).—See B.P. 267,235; B., 1927, 538.

Crystalliser [for sugar solutions]. F. LAFEUILLE (U.S.P. 1,653,712, 27.12.27. Appl., 10.7.25. Fr., 24.12.24).—See B.P. 245,114; B., 1926, 992.

XVIII.—FERMENTATION INDUSTRIES.

Physiology of *Bacillus acidificans longissimus* (*B. delbrücki*) in connexion with its possible use in the production of lactic acid. V. SHAPOSHNIKOV and A. V. MANTEIFEL (Trans. Sci. Chem. Pharm. Inst., 1923, No. 7, 3—23).—On the second day neutralisation is effected with magnesium oxide, magnesium carbonate, or zinc oxide. CHEMICAL ABSTRACTS.

Thermophilic species of *Penicillium arenarium* nov. sp. producing citric acid. V. SHAPOSHNIKOV and A. Y. MANTEIFEL (Trans. Sci. Chem. Pharm. Inst., 1923, No. 5, 3—27, 28—56).

Determination of pepsin. HOFFMANN.—See XX.

PATENTS.

Cooling device for use in the quick vinegar process. A. STEINMETZ (G.P. 443,802, 25.9.25).—The upper part of the apparatus is provided with straight or bent hollow plates, through which cooling water circulates, arranged to divide the space filled with the shavings into small compartments. L. A. COLES.

Cultivation of edible fungi (B.P. 270,684).—See XVI. **Lacto-drinkables** (B.P. 266,692).—See XIX.

XIX.—FOODS.

Variations in the proportion of solids-not-fat in milk. J. F. TOCHER (Scottish J. Agric., 1927, 10, 201—210).—Average, minimum, and maximum values were 8.80, 7.5, and 10.75%, respectively; average values for Ayrshire, Friesian, and other cows were 8.75, 8.62, and 8.86%, respectively. The daily values from a herd fluctuated between 8.4 and 9.7%. The values, which were minimal about 20 weeks after calving, increased with the yield and with the age of the cow.

CHEMICAL ABSTRACTS.

Non-protein nitrogen in certain dairy rations and partition of nitrogen in the urine produced thereon. W. E. KRAUSS (J. Dairy Sci., 1927, 10, 400—415).—The distribution, digestibility, and utilisation of the non-protein nitrogen in dairy rations was investigated. Rations composed of hay, silage, and grain contain a relatively large amount of non-protein nitrogen. CHEMICAL ABSTRACTS.

[Cow's milk.] M. H. CAMPBELL, M. J. PRUCHA, and J. M. BRANNON (Illinois Sta. Rep., 1926, 93—96).—A method for determining the size of fat globules is given. Edestin feeding did not increase the rate of milk secretion. The preparation of acidophilous milk is described.

CHEMICAL ABSTRACTS.

Addition of calcium chloride to milk for cheese-making. W. V. PRICE (J. Dairy Sci., 1927, 10, 373—376).—In presence of calcium chloride the action of rennin is more rapid; the coagulum is firmer and the loss of moisture after cutting is more rapid, although at the time of pressing the moisture content was normal.

CHEMICAL ABSTRACTS.

Increasing the yield of cheese by the addition of calcium chloride to milk. G. KNAYSI and J. D. NELSON (J. Dairy Sci., 1927, 10, 396—399).—Treatment of cheese with (anhydrous) calcium chloride before the addition of rennin increases the yield and reduces the amount of rennin necessary. There is also more complete precipitation of phosphate and calcium.

CHEMICAL ABSTRACTS.

Determination of age of eggs by means of ultra-violet fluorescence. J. E. H. VAN WAEGENINGH and J. E. HEESTERMAN (Chem. Weekblad, 1927, 24, 622—623).—The white of perfectly fresh eggs shows no fluorescence in ultra-violet rays, but, on keeping, a fluorescence develops and becomes stronger with time. The age of eggs may be determined by comparing the fluorescence with that given by gelatin solutions between 0.1 and 10% strength. At 16 days the fluorescence is equal to that of a 0.25% solution, and at 120 days to that of a 12.5% solution.

S. I. LEVY.

Fruit jellies. V. Rôle of pectin. 1. Viscosity and jellifying properties of pectin solutions. P. B. MYERS and G. L. BAKER (Univ. Del. Agric. Exp. Sta. Bull., 1927, No. 149).—The increase in viscosity of pectin solutions on increase of acidity is negligible in comparison with that caused by the addition of alkali. No indication of the isoelectric point of pectin is obtained by means of viscosity measurements. Jelly strength is a function of the viscosity of the pectin solution, and is independent of its concentration. The p_H at the optimum point of jelly formation varies with the concentration of pectin. The calcium pectate method for determining the jellifying power of a pectin is not considered trustworthy. Precipitation of pectin by ethyl alcohol is preferably effected by dropwise addition and constant stirring; prolonged heating is deleterious. Pectins obtained from pectin substances by extraction with distilled water are not necessarily of a better quality than those extracted with acid.

CHEMICAL ABSTRACTS.

Viscosity of egg-white. JOCHIMS.—See I. **Cacao-red.** HEIDUSCHKA and BIENERT.—See IV.

PATENTS.

Treatment of meal, flour, or other milling products. J. W. DOMBACH (B.P. 258,279, 10.9.26. Ger., 10.9.25).—The products from individual units of a milling plant are grouped according to their properties, and each group is separately treated, as required by its particular chemical and physical properties ascertained by test, by moistening, bleaching, dressing, or addition of chemical substances so as to improve the quality.

After further separate treatment in the milling process, the groups are mixed to form the finished product.

F. R. ENNOS.

Production of cereal and other food products. V. D. ANDERSON Co., Assees. of R. T. ANDERSON (B.P. 278,653, 19.3.27. U.S., 9.10.26).—Food, e.g., flour, is subjected to a gradually increasing pressure (5000—10,000 lb./in.²) whereby it is heated and partly cooked. Water is then forced into the compressed and heated mass, and the steam generated serves to complete the cooking process; the product is finally extruded from the press in any desired form.

F. R. ENNOS.

Manufacture of bread and other cereal baked foods. W. D. STEIN (B.P. 281,979, 28.9.26).—Flour is mixed with the product containing dextrinised starch acid esters, obtained by treating starch with acetic acid and heating, and the mixture is made into dough by addition of the usual ingredients, except sugar, and baked.

F. R. ENNOS.

Manufacture of butter and apparatus therefor. J. JOSEFSON, Assee. of T. A. WIBERG (B.P. 262,769, 4.12.26. Swed., 14.12.25).—Butter is produced in a continuous operation by feeding cream or whole milk between a rapidly rotating dasher and a stationary casing surrounding it, the intervening space being of considerable width where the material is first treated, but becoming constricted at the part through which the churned mass flows towards the outlet.

F. R. ENNOS.

Obtaining the total bodies containing phosphorus and iron which are derived from the proteids of egg yolk. S. POSTERNAK (B.P. 268,805, 31.3.27. Switz., 31.3.26).—The extract obtained by treating egg yolk with boiling alcohol is dried, ground, and subjected to pepsin digestion in the presence of mineral acid. After a predetermined time the reaction is arrested by addition of sodium carbonate or acetate, and the precipitated proteids are collected on a filter and subjected to trypsin digestion in a slightly alkaline solution, whereby the substances containing phosphorus are split off. The liquid is then acidified and filtered, and the mixture of the substances containing phosphorus is obtained from the filtrate as alkaline-earth salts by treatment with a soluble alkaline-earth salt and alcohol, or as alkali salts by precipitation and decomposition of the corresponding heavy-metal salts, the free acids as well as all other salts being obtained from these by the usual methods.

F. R. ENNOS.

Production of sour lacto-drinkables. S. MATSUBARA (B.P. 266,692, 9.2.27. Japan, 27.2.26).—Milk, after being sprayed into the air through a small nozzle, is heated below 70° for about 30 min. to remove odours and ensure sterilisation, and is then subjected to lactic fermentation for about 30 hrs. by means of *B. lacticum* until it contains 1.15% of lactic acid. A concentrated solution of sugar is then added, and the whole is cooled to 15° and thoroughly mixed by subjecting it to high-compressive shocks by forcing it, under air pressure of at least 5000 lb./in.², through an exceedingly small gap formed between a valve and its well-ground seating.

F. R. ENNOS.

Preservation of fruits etc. R. V. JELICOF (B.P. 281,380, 31.8.26).—The syrup or other preserving fluid

is made to circulate continuously in a closed path through the fruit etc. which is supported on a series of collapsible perforated trays in a heated, evacuated container. The concentration of the syrup is determined at intervals by measuring the volume of the condensed vapour from the container, and when the operation is complete the residual syrup is pumped off and the impregnated fruit removed.

F. R. ENNOS.

Manufacture of pectin products. SCHWARTAUER HÖNIGWERKE U. ZUCKERRAFFINERIE A.-G. (B.P. 281,513, 1.3.27. Ger., 1.2.27).—Fruit or vegetable material is alternately lixiviated with cold, acidified water and pressed out until the water drained off contains none of the constituents soluble in cold water. The material is then repeatedly extracted with hot solutions of tartaric or similar organic acid, and the fractions obtained, after clarifying and bleaching if desired, are either concentrated or dried under vacuum separately or together. Preferably the non-gel-forming pectin is first separated by filtration and the gel-forming pectin substances remaining on the filter are colloiddally dissolved to any desired concentration or are dried under vacuum, the p_H value of the concentrated solutions being so adjusted, by addition of suitable organic acids, that gelatinisation may occur.

F. R. ENNOS.

Coagulation or curdling of milk. H. A. KOHMAN, R. IRVIN, and E. S. STATELER, Assrs. to G. S. WARD (U.S.P. 1,654,176, 27.12.27. Appl., 3.10.21).—See B.P. 186,923; B., 1923, 794 A.

Edible fungi (B.P. 270,684).—See XVI.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Removal of peroxides from anæsthetic ether. J. RAE (Pharm. J., 1927, 119, 693—694).—Ether which gave a very strong reaction for peroxides gave no reaction after being kept, with frequent shaking, in contact with powdered animal charcoal for 54 days.

E. H. SHARPLES.

Determination of pepsin. K. HOFFMANN (Pharm. Ber., 1927, 2, 45—47; Chem. Zentr., 1927, i, 2674).—Bachstetz's modification of Gross' method was employed. Various commercial pepsin preparations had scarcely any activity.

A. A. ELDRIDGE.

Causes of precipitation in tinctures and fluid-extracts. W. L. SCOVILLE (J. Amer. Pharm. Assoc., 1927, 16, 1136—1146).—Official aqueous-alcoholic tinctures of alkaloidal, astringent, cathartic, and miscellaneous drugs were treated with various agents (light, hydrochloric acid, sodium chloride, ammonia, hydrogen peroxide, hypophosphorous acid, carbon dioxide, and sodium acetate), in order to determine the causes of precipitation. Precipitation is mainly due to hydrolysis, and may be restrained by increasing the concentration of the alcohol.

B. FULLMAN.

Reactions of atophan and novatophan. L. EKKERT (Pharm. Zentr., 1927, 68, 797).—Both compounds in hydrochloric acid solution give red colorations with α - or β -naphthol.

S. I. LEVY.

Analysis and characterisation of neosalvarsan-amine [neosalvarsan]. A. E. JURIST and W. G. CHRISTIANSEN (J. Amer. Pharm. Assoc., 1927, 16, 1149—1152).

—The method of Elvove (B., 1926, 27) for the examination of neosalvarsan is modified in that, after oxidation with iodine in neutral solution, the excess of iodine is reduced with excess of sodium arsenite. The resultant sulphate sulphur is determined in acid solution as barium sulphate. The sulphosalvarsan sulphur of Elvove is really nuclear sulphur introduced in the preparation of the base from which the neosalvarsan is made. Factors are given for the determination of the following constituents of neosalvarsan: free sulphonylate, mono- and di-substituted neosalvarsan, free base, and sodium sulphate. The examination of numerous specimens of neosalvarsan from this point of view reveals the presence of a form of sulphur of unknown nature, oxidised by iodine only in strongly alkaline solution. Specimens of neosalvarsan may be classified in four groups according to the proportion present of mono- and di-substituted neosalvarsan, free sulphonylate, and non-sulphonylate sulphur.

B. FULLMAN.

Ethereal oils extracted from the principal native aromatic plants of Erythrea. P. ROVESTI (Annali Chim. Appl., 1927, 17, 553—570).—The seeds of *Carum chitium*, Benth., known as *azmuti* in Erythrea and as *camum* in Tigris, contain 3.65% of an orange oil of thymolic odour and burning taste, having d_{15}^{20} 0.9252, $\alpha_D^{20} + 1.47^\circ$, n_D^{20} 1.5085, solubility in 80% alcohol 1:1.3 at 25° ; it contains 52% by volume of crystallisable thymol, together with *d*- α -pinene and *p*-cymene, but is free from carvacrol. The dried leaves, stems, and flowers of *Lippia adoensis*, Hochst., contain 0.753% of an orange-yellow oil having d_{15}^{20} 0.93395, $\alpha_D^{20} + 55.3^\circ$, n_D^{20} 1.4958, solubility in 75% alcohol 1:2.6 at 20° , and containing *d*-limonene and about 72% by vol. of carvone. *Andropogon comatus*, Hochst., yields 1.78% of a yellowish oil resembling Indian gingergrass oil, having d_{15}^{20} 0.9355, $\alpha_D^{20} - 54.5^\circ$, n_D^{20} 1.4951, solubility in 70% alcohol 1:2.1 at 20° , and containing a small proportion of an aldehyde of penetrating odour, α -phellandrene, geraniol, 30—35% of dihydrocumyl alcohol, and other hydrocarbons and sesquiterpenic compounds. *Ocimum menthaefolium*, Hochst., yields 0.327% of an almost colourless, pleasant-odoured oil, having d_{15}^{20} 0.9496, $\alpha_D^{20} + 1.4^\circ$, n_D^{20} 1.5165, solubility in 80% alcohol 1:3.1 at 25° , and composed mostly of methylchavicol, anethole, small proportions of free and esterified alcohols, hydrocarbons, and sesquiterpenes. *Meriandra benghalensis*, Benth., gives a yellow, semi-solid oil, d_{15}^{20} 0.9318, $\alpha_D^{20} - 6.2^\circ$, n_D^{20} 1.4687, solubility in 70% alcohol 1:2.1 at 20° , which furnishes a yield of 70—72% of camphor and a residual product capable of replacing Dalmatian or Spanish rosemary oil in soap-making.

T. H. POPE.

Esters of lemon and orange oils. H. R. JENSEN (Perf. Ess. Oil. Rec., 1927, 18, 510).—The ratio of aldehydes to esters (expressed as linalyl acetate) should be about 2.5 when the latter are determined by saponification with 0.5*N*-alcoholic potassium hydroxide and boiling for 15 min. only. Higher ratios indicate adulteration with citral.

S. I. LEVY.

Odoriferous principle of castoreum. H. WALBAUM and A. ROSENTHAL (J. pr. Chem., 1927, [ii], 117, 225—232).—Steam distillation of the alcoholic extract of castoreum yields an oil (2.1%) containing acetophenone, benzyl

alcohol, *l*-borneol, *p*-ethylphenol (α -*naphthyl*carbimide derivative, m.p. 128°), *o*-cresol or *o*-ethylphenol, either guaiacol or creosol, and a small quantity of benzoic acid.

H. BURTON.

PATENTS.

Calcium-sugar preparations. [A] H. A. SHONLE, [B] G. VAN SCOYOC and H. L. WEHRBEIN, [C] G. VAN SCOYOC, H. L. WEHRBEIN, and H. A. SHONLE, Assrs. to E. LILLY & Co. (U.S.P. 1,649,000, 1,649,269, and 1,649,270, 15.11.27. Appl., [A] 9.1.26, [B, C] 1.5.24).—(A) Dextrose solution is heated with calcium hydroxide until the greater portion of the latter has combined with the acid degradation products of the sugar. The solution is then neutralised with oxalic acid to remove excess of calcium. The filtrate from the precipitated calcium oxalate is of therapeutic value. (B) The addition of oxalic acid is omitted. (C) The total calcium in the solution is removed with oxalic acid. The filtrate, which is more stable than that obtained in (A), is treated with calcium hydroxide solution before use.

T. S. WHEELER.

Manufacture of β -amino- β' -dialkylaminoisopropyl alcohols. I. G. FARBENIND, A.-G. (B.P. 276,012, 12.8.27. Ger., 13.8.26).— β' -Halogeno- β -aminoisopropyl alcohols, $X \cdot CH_2 \cdot CH(OH) \cdot CH_2 \cdot NH_2$, or the corresponding phthalimides, $X \cdot CH_2 \cdot CH(OH) \cdot CH_2 \cdot NC_2O_2 : C_6H_4$, are treated with a secondary amine. γ -Chloro- β -hydroxypropylphthalimide reacts with diethylamine to give, after hydrolysis with acid, β -amino- β' -diethylaminoisopropyl alcohol, b.p. 114–115°/20 mm., also obtained from β' -chloro- β -aminoisopropyl alcohol hydrochloride. The diaminoalcohols reduce the amount of sugar in the blood, and are intermediates for other therapeutic compounds.

C. HOLLINS.

Production of active sedative, hypnotic, and sleep-producing compounds, and compounds produced by such process. A. E. VIDAL. From J. D. RIEDEL A.-G. (B.P. 281,365, 27.8.26).—Acids of the type, $CH_2 : CH \cdot CH_2 \cdot CHR \cdot CO_2H$, in which R = alkyl or alkylenyl, are converted by the usual methods into ureides useful as sedatives and hypnotics. α -iso-Propyl- Δ^1 -pentenoyl chloride, b.p. 63–64°/15 mm., obtained from the acid by the action of thionyl chloride, gives with carbamide a ureide, m.p. 190–191°, obtainable also from the acid amide, m.p. 107°, and cyanic acid. Hydrolysis and decarboxylation of ethyl allylbutylmalonate yields α -allylhexoic acid, b.p. 225–230° (chloride, b.p. 60°/12 mm.), from which the ureide, m.p. 147–148°, is prepared.

C. HOLLINS.

New therapeutic substances. WELLCOME FOUNDATION, LTD., T. A. HENRY, and T. M. SHARP (B.P. 281,582, 16.8.27).—Dilute hydrochloric acid extracts from the alcoholic extract of the fat-freed, ground seeds of *Picralima Klaineana* (Pierre) a mixture of three alkaloids, the free bases being precipitated by alkali carbonates. For the separation of the alkaloids the solution of the hydrochlorides is diluted with water until no further precipitation of alkaloid-A occurs. The remaining alkaloids, after precipitation with sodium carbonate, are extracted with dry ether, which leaves a further amount of alkaloid-A undissolved. The extract is evaporated to dryness and the dry residue macerated with twice its weight of cold alcohol; alkaloid-B goes

into solution, leaving alkaloid-C undissolved. Alkaloid-A after purification in boiling alcohol has m.p. 248°. Alkaloid-B is obtained from the above alcoholic solution by evaporation, dissolution in acid, and reprecipitation by carbonate; it has m.p. 140° (air-dry) or 167° (dried in a vacuum). Alkaloid-C is purified as sulphate, $B_3 \cdot 2H_2SO_4$, m.p. 221°, the free base, $C_{22}H_{28}O_4N_2$ (hydrochloride, $B \cdot HCl \cdot H_2O$, m.p. 227°, $[\alpha]_D^{20} - 26.6^\circ$; hydrobromide, $B \cdot HBr \cdot H_2O$, m.p. 228°, $[\alpha]_D^{20} - 26.05^\circ$), being precipitated with carbonate. The alkaloids, separately or as mixture, are used against malaria.

C. HOLLINS.

Manufacture of an ergot preparation. G. ERDMANN, Assr. to GEHE & Co. A.-G. (U.S.P. 1,645,096, 11.10.27. Appl., 28.5.26. Ger., 18.3.25).—Ergot is extracted with ether at ordinary temperature and the extract is treated with low-boiling petroleum hydrocarbons. The precipitate thus obtained is dissolved in dilute acetic acid, and, after washing with ether, the solution is precipitated with ammonia. Alternatively, the material extracted from the crude drug with ether is washed with ether containing an acid, and then treated with an alkali solution. The bases thus precipitated are dissolved in chloroform and reprecipitated with low-boiling petroleum hydrocarbons. T. S. WHEELER.

Arsenising of organic compounds. E. SCHELLER (B.P. 261,026, 5.11.26. Austr., 9.11.25).—Aromatic or heterocyclic compounds, prepared in glacial acetic acid, methyl alcohol, etc. in the absence of much water, react with arsenic halides (arsenic trichloride) to give products which are reduced by sodium hyposulphite to arseno-compounds, from which arsenic acids are obtained by oxidation. The arsenic halide may be present during diazotisation.

C. HOLLINS.

Manufacture of a [non-toxic] medical preparation containing arsenic. I. OSTROMISLENSKY, Assr. to OSTRO PRODUCTS CORP. OF AMERICA (U.S.P. 1,644,348, 4.10.27. Appl., 22.7.22).—The toxicity of arsenic compounds, e.g., phenylarsenious oxide, is reduced by dispersion in an aqueous solution containing a protective colloid, e.g., gum arabic, and, preferably, dextrose in quantity sufficient to render it isotonic with blood.

T. S. WHEELER.

Manufacture of water-soluble, neutral [lactose] derivatives of diaminodihydroxyarsenobenzene. H. M. WUEST, Assr. to HOFFMANN-LA ROCHE CHEMICAL WORKS (U.S.P. 1,648,926, 15.11.27. Appl., 28.5.23. Switz., 3.6.22).—Diaminodihydroxyarsenobenzene is dissolved in lactose solution, and the mixture is treated with alcohol to precipitate a relatively non-toxic product of therapeutic value and stable out of contact with air.

T. S. WHEELER.

Concentration of [radio]active deposit. H. B. PALMER (U.S.P. 1,644,350, 4.10.27. Appl., 15.4.25).—Pills containing non-toxic conducting material, e.g., carbon, are negatively charged and subjected to the action of niton immediately before use. T. S. WHEELER.

Preparation of phosphatides. A. GRÜN and R. LIMPÄCHER, Assrs. to G. SCHICHT A.-G. (U.S.P. 1,653,959, 27.12.27. Appl., 23.9.24. Czechoslov., 27.9.23).—See B.P. 222,463; B., 1925, 941.

Production of halogeno-albumin compounds. R. WOLFFENSTEIN (U.S.P. 1,649,051, 15.11.27. Appl., 2.2.24. Ger., 29.3.23).—See B.P. 213,536; B., 1924, 926.

Preparation of aliphatic di- or poly-hydroxy-arsinic acids. C. J. OECHSLIN (U.S.P. 1,654,224, 27.12.27. Appl., 26.1.25).—See B.P. 191,028; B., 1923, 861 A.

Benzoxazolonearseno-compounds. L. BENDA, Assr. to I. G. FARBEIND. A.-G. (U.S.P. 1,648,214, 8.11.27. Appl., 2.6.24. Ger., 9.6.23).—See B.P. 239,951; B., 1925, 942.

Ampulla for the preparation of solutions, more particularly for medical and like purposes. PHARMAGANS PHARMACEUT. INST. L. W. GANS A.-G. (B.P. 280,838, 22.7.26. Addn. to B.P. 264,125; B., 1928, 37).

Substituted diaminopropanols (B.P. 275,622).—See III.

Insecticides etc. (B.P. 280,256).—See XXIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Intensification of the latent image on photographic plates and films. II. Decomposition of hydrogen peroxide and the mechanism of latent image intensification. E. P. WIGHTMAN and R. F. QUIRK (J. Franklin Inst., 1927, 204, 731—749; cf. B., 1927, 268).—Alkaline hydrogen peroxide in presence of gelatin-silver or gelatin-silver sulphide sol, with or without the presence of soluble bromide, is more easily decomposed than acid peroxide under the same conditions. Both acid and alkaline peroxide in the presence of soluble bromide react with silver sulphide in bulk to give sulphuric acid or a sulphate and colloidal silver bromide. Colloidal silver dissolves to a greater or less extent in gelatin-hydrogen peroxide solution with a p_H value of 5.5 or less, giving rise to silver ions which, when soluble bromide is also present, form colloidal silver bromide. These facts indicate that intensification of the latent image, and probably also the effect of peroxide on sensitivity or the production of latent fog, is chemical in nature as Clark has contended (B., 1926, 253), and that a chemiluminescence mechanism is not necessary to explain the phenomenon. A chemical mechanism is proposed, based on Hickman's theory of latent image formation. It is suggested that the bromine set free by the action of acid peroxide on the soluble bromide in the plate, being very limited in quantity, acts on the silver sulphide speck to form metallic silver, which in turn may be partly converted by the acid peroxide into silver ions, and if any soluble bromide is in the vicinity some colloidal silver bromide is formed containing on its surface adsorbed silver ions. It is believed that any one or all of these, metallic silver, silver ions, or colloidal silver bromide with silver ions adsorbed, produces a greater degree of developability of the grain than the original silver sulphide speck. The view is supported by the observation that if soluble bromide is largely removed from a plate by the action of silver nitrate (less than excess), hydrogen peroxide produces very little, if any, intensification of the latent image.

W. CLARK.

The photographic emulsion and its application in research. H. STINTZING (Z. angew. Chem., 1927, 40, 1423—1431).—A summary of the published work on the properties of the unexposed and the developed emulsion, including ripening grains and nuclei, the crystalline properties of the silver bromide grain, grain-size distribution, the latent image and the print-out image, physical development, sensitivity, gradation, resolving power, turbidity, etc.

W. CLARK.

Newer photographic problems. A. STEIGMAN (Kolloid-Z., 1927, 43, 400—402).—The photographic quality of gelatin is not defined simply by the content of sulphur which answers to the lead test. The degree of lability of the sulphur in the organic sensitizers or mixtures of desensitisers is of importance. Organic desensitisers also have an influence on the photographic quality, as is known to be the case with inorganic desensitisers. Different silver salts take the sulphur from thiosinamine at different rates at p_H 5.6. Serum albumin, added to gelatin before mixing, in the case of a chlorobromide emulsion, is a strong desensitizer and prevents ripening. The wash water from a good ripening gelatin also desensitises, as do also cystine and tyrosine. The action of cystine is probably due to its desensitising power.

W. CLARK.

PATENT.

Preparation of [non-phosphorescent] fluorescent material [for X-ray screens]. S. E. SHEPPARD, Assr. to EASTMAN KODAK CO. (U.S.P. 1,648,510, 8.11.27. Appl., 2.2.26).—The addition of sodium sulphide (0.5—2%) to the calcium tungstate used in the manufacture of intensifying X-ray screens inhibits phosphorescence of the screen.

T. S. WHEELER.

XXII.—EXPLOSIVES; MATCHES.

Laws of combustion of colloidal [explosive] powders. III. H. MURAOUR (Bull. Soc. chim., 1927, [iv], 41, 1451—1461; cf. B., 1926, 854).—Experiments on the combustion in a closed vessel of colloidal explosive powders with widely different explosion temperatures have shown that the rate of combustion, V , is related to the pressure, P , by the equation $V = a + bP$, where a and b are constants for a particular powder, and a is negligible at high pressures. At high pressures, the relation $V = Pe/K$ is approximately true, e being the thickness of the threads of explosive, and K the coefficient defined previously (B., 1926, 722). With decrease in the heat of combustion of the powder, the rate of combustion at a given pressure decreases also, but more rapidly.

R. CUTHILL.

Denitration of waste acids. SCHMID.—See VII.

PATENTS.

Detonator tube. W. ESCHBACH (G.P. 443,727, 1.8.26).—Iron coated with aluminium is used in the manufacture of detonator tubes. These tubes are unacted on by the detonator composition and have the additional advantage, as compared with aluminium tubes, of being more suitable for use in mines containing firedamp.

S. BINNING.

Liquid-air blasting cartridge. RUSSFABR. KAHL (G.P. 440,233, 1.2.25).—Sulphur-containing organic compounds, which may be solid (vulcanised rubber or

factis) or liquid (solutions of sulphur in linseed or turpentine oils), are added to the absorbent material. The life and strength of the cartridge are thereby increased. S. BINNING.

Obtaining nitrocellulose from smokeless powder. J. K. SPEICHER, Assr. to HERCULES POWDER Co. (U.S.P. 1,653,519, 20.12.27. Appl., 4.2.27).—Smokeless powder containing diphenylamine is dissolved in acetone, and the solution introduced into toluene heated to the b.p. of acetone. The nitrocellulose is recovered from the toluene. B. FULLMAN.

Production of waterproofing composition [for explosives]. T. A. O'BARR, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,648,861, 8.11.27. Appl., 8.3.24).—A mixture of a vegetable drying oil, *e.g.*, tung oil, a resin, *e.g.*, colophony, and paraffin hydrocarbons, *e.g.*, petrolatum, is used as a waterproof coating for ammonium nitrate explosives. T. S. WHEELER.

XXIII.—SANITATION ; WATER PURIFICATION.

Effect of chlorination on the digestion of sewage solids. G. M. FAIR and C. L. CARLSON (Eng. News-Rec., 1927, 99, 1030—1031).—Chlorine introduced into sewage as a disinfectant or deodorant prior to the separation of suspended solids is shown by laboratory-scale experiments to favour the production of a sludge which is capable of digestion at an increased rate as measured by the rate of evolution of methane. C. JEPSON.

Chlorination of screened sewage at Bridgeport, Conn. W. J. SCOTT and C. L. POOL (Eng. News-Rec., 1927, 99, 1033—1034).—Satisfactory reduction in bacterial content of this sewage after fine screening required the addition of 10—15 p.p.m. of chlorine and a contact period of 10 min. Careful chlorine control using the *o*-tolidine test so as to maintain a residual of 0.3 p.p.m. is necessary for economic application. C. JEPSON.

Odour control [of crude sewage] by chlorination at Independence, Kan. G. H. HACKMASTER (Eng. News-Rec., 1927, 99, 1031—1033).—Aerial nuisance due to proximity of sewage treatment plant has been effectively prevented by the addition of chlorine to the sewage in doses varying from 3 to 10 pts. per million, when weather conditions favoured the nuisance. The average dose over a 56 day period was 3.32 p.p.m., and the average cost was \$3.32 per million gals. It was found more effective to add chlorine before rather than after separation of suspended solids. C. JEPSON.

Five years of rapid sand filtration [of water] at Cambridge, Mass. M. C. WHIPPLE and H. C. CHANDLER (J. New England Water Works' Assoc., 1927, 41, 218—243).—The raw water, which is derived from several sources, is coloured with extract of dead vegetation, dull in appearance, impregnated with odours and tastes from dead and living plants, at times distinctly corrosive towards metals, and liable to entrance of germs of water-borne diseases. Purification comprises coagulation with sodium aluminate and aluminium sulphate, rapid filtration, aeration, corrective treatment to eliminate corrosive qualities, and chlorination. Coagulation demands careful study and regulation

owing largely to the changes in quantity and age of the organic matter contained in the water. Alum alone was formerly used as coagulant, but combined sodium aluminate (0.17 grain/gal.) and alum (0.8—1.25 grains/gal.) treatment is now preferred as it results in a treated water of lower corrosive properties. The cost of the combined treatment is slightly greater, but a substantial saving is effected in the amount of soda ash used for corrective treatment. Repeated observations have failed to indicate any advantage to be derived from chlorination of the raw water prior to coagulation. Hydrogen-ion concentration control for coagulation is not adequate to ensure the best results on all occasions. Rates of filtration range from 118—136 million gal. per acre per day. Mud balls are eliminated by occasional caustic soda treatment. The average colour of the raw water, settled water, and final effluent is 42, 31, and 4 p.p.m., respectively. The average numbers of *B. coli* in raw, settled, and filtered water, and final effluent are 10, 0.6, 0.1, nil per 100 c.c. samples. W. T. LOCKETT.

Eliminating a source of error in the colorimetric determination of manganese. A. C. JANZIG (J. Amer. Water Works' Assoc., 1927, 18, 744—745).—Minute traces of manganese may be accurately determined, even in highly coloured or turbid waters, using the persulphate method described in "Standard Method of Water Analysis" (Amer. Public Health Assoc.), provided an excess of ammonium persulphate is maintained during the removal on a filter of insoluble matter which interferes with the comparison of the colours produced. C. JEPSON.

PATENTS.

Accelerating the sludge digestion for sewage treatment. M. PRÜSS (B.P. 281,541, 23.5.27).—The sludge contained in digestion chambers is circulated periodically by drawing it through a pipe (by means of a pump or other suitable appliance) from the bottom of the digestion chamber and discharging it near the surface level. By this process, gases, *e.g.*, carbon dioxide, which are injurious to digestion and are dissolved in the sludge water at the bottom part of the digestion chamber, are eliminated according to the decrease in hydraulic pressure, and injurious liquid decomposition products are removed from the particles of sludge and diffused throughout the sludge water. By a reversal of the process, floating sludge may be drawn off from the surface and forced into the bottom part of the digestion chambers, and in consequence of the admixture with active settled sludge and impregnation with fresh active bacteria it becomes more easily decomposed. W. T. LOCKETT.

Insecticides, sheep-dips, etc. McDougall & YALDING, LTD., and P. J. FRYER (B.P. 280,256, 24.11.26).—The water-insoluble, physiologically active extracts or resins, particularly tuba-toxin and tephrosin, from leguminous plants, *e.g.*, derris, are dissolved in a phenol (cresylic acid) and mixed with hydrocarbon oil from mineral oil or from coal tar (preferably a medium lubricating oil fraction of American petroleum), and the whole is emulsified with water in presence of soap, glue, casein, etc. (cf. B.P. 229,773; B., 1925, 334). C. HOLLINS.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 2, 1928.

I.—GENERAL; PLANT; MACHINERY.

Small high-pressure autoclave. F. N. PETERS, JUN., and O. C. STANGER (Ind. Eng. Chem., 1928, 20, 74—76).—The autoclave is designed for the agitation of a liquid with hydrogen at 2000 lb./sq. in. pressure and temperature up to 200°. The shell consists of steel pipe with a T-piece and cap on one end, these joints being welded. The other end is machined and grooved and closed by a steel disc with lead gaskets. The hydrogen supply is connected to the T-piece by copper tube. Within the shell is a reaction tube with central opening, and agitation is effected by rocking longitudinally—a method which is very efficient. The shell is surrounded by asbestos cement insulation and provided with heating elements. C. IRWIN.

Apparatus for storing and circulating gases [in the laboratory]. A. H. WHITE and F. W. HIGHTOWER (Ind. Eng. Chem., 1928, 20, 95—97).—Two rubber balloons are each contained in an inverted bottle filled with water. These bottles are connected to each other and also to a higher water reservoir which rests upon one beam of a balance. Gas can be pumped from one balloon to the other. Any change in gas volume is immediately indicated and measured by the alteration of the water content of the reservoir. The pump used was of the mercury piston type, the mercury being operated by air pressure from a cycle pump geared to a motor. The apparatus provides a means of studying gas reactions without the use of any large quantity of mercury. The principal error arises from diffusion of gases through rubber, which is not serious during a few hours' work. C. IRWIN.

Gas mask. HEINRICH and PETZOLD.—See XXIII.

PATENTS.

Firing of furnaces with pulverised fuel. F. H. ROSENCRANTS (B.P. 281,850, 12.11.26).—For furnaces having fuel pulverisers directly connected to them, a slow-speed mill provides fuel sufficient for minimum load, and a high-speed mill that for the variable increase. A small variation in the speed of the first mill may be obtained by altering the speed of its motor.

B. M. VENABLES.

Rotary hearths for furnaces. C. NELSON, and KING, TAUDEVIN, & GREGSON, LTD. (B.P. 282,135, 9.9.26).—The rotary hearth is supported on a ball bearing and driven by a worm, the whole mechanism being submerged in a shallow oil bath. The upper ball race is fixed to the hearth, but the lower is free to accommodate itself horizontally on the bottom of the bath. The hearth is centred horizontally by an axial pivot which is free vertically. B. M. VENABLES.

Heat-transmitting tubes. W. J. STILL (B.P. 282,244, 16.9.26).—A description is given of machines for winding wire on the outside of tubes to increase the heat-transmitting surface. B. M. VENABLES.

Pulverisers. H. WARING, and ASSOCIATED LEAD MANUFACTURERS, LTD. (B.P. 282,145, 13.9.26).—A high-speed disintegrator for soft and fibrous material.

B. M. VENABLES.

[Grinding body for] tube and drum mills. E. C. R. MARKS. From G. POLYSIUS (B.P. 281,528, 13.4.27).—The ends of a helical spring are bent across the axis, and in the space so enclosed are placed one or more loose cylinders or balls. A number of these elements form the ball charge of the mill. B. M. VENABLES.

Apparatus for stirring and mixing. J. Y. JOHNSON. From I. G. FARBERIND, A.-G. (B.P. 281,852, 15.11.26).—In a vessel the contents of which are agitated by a propellor with inclined or helical blades, other fixed blades are used of opposite inclination to the moving ones, and so arranged that the circulating liquid passes through the fixed guides first.

B. M. VENABLES.

Mixing and stirring apparatus. F. A. EASTERBROOK, and BROWN & SON (ALEMBIC WORKS), LTD. (B.P. 281,904, 25.2.27).—A form of mixing propellor suitable for the reconstitution of dried milk and similar purposes.

B. M. VENABLES.

Apparatus for drying. A. F. WRIGHT (B.P. 281,785, 14.9.26).—A drying atmosphere is circulated by a fan through drying chambers, which alternate with re-heating chambers, to a conditioning chamber where a proportion of fresh air is admitted, back to the fan. The exhaust air, fresh air, and recirculated air are controlled by separate valves, and the re-heaters are controlled by a thermostat in the return air duct.

B. M. VENABLES.

Centrifugal dryers and separators. A. HABERMANN (B.P. 265,561, 5.1.27).—The apparatus comprises a perforated drum—preferably conical—and a separately driven internal worm, both of which revolve at high speed, but have a slow relative motion by which, in conjunction with longitudinal ridges on the interior of the drum, the dried material is conveyed to the outlet end of the drum. A cylindrical extension of the drum carries scrapers to remove any discharged material adhering to the outer casing. B. M. VENABLES.

Centrifugal separator and dryer. P. FESCA (C. A. FESCA & SOHN) (B.P. 278,327, 15.8.27. Ger., 1.10.26).—In a centrifuge in which the basket comprises two cones base to base, held together when separating liquid from solids, and separated when discharging the collected.

solid, the two portions are held together by a hydraulic cylinder and piston within the basket.

B. M. VENABLES.

Centrifugal machine. H. J. GREAVES (B.P. 281,483, 16.12.26).—The basket of a centrifugal separator is formed of a number of channel-shaped, vertical slats through which the liquid is filtered off continuously. Within and without the basket are shells rotating together at a very slightly different speed from that of the basket. The inner shell is provided with ports for supplying feed to the basket and the outer shell with an equal number of ports for discharge of solid matter. The same gearing which causes the slow differential speed also overturns the slats of the basket in turn when they are opposite the discharge ports. The inlet ports are always just behind the discharge ports.

B. M. VENABLES.

Apparatus for separating solid materials by the float and sink method. C. N. KEMP and J. L. THOMSON (B.P. 281,479, 13.12.26).—The material to be separated is placed in a cylindrical basket with perforated bottom, which is then placed in a cylindrical tank. Liquid of high sp. gr. is allowed to enter the tank from the bottom. The float is skimmed off by a perforated scoop which normally lies in a recess at the side of the tank. After the scoop has been placed under the float the liquid is drained off, and not till then is the scoop lifted. Thus all splashing and waste of liquid are avoided. The process may be repeated with heavier liquids in succession. (Cf. B., 1927, 641.)

B. M. VENABLES.

Separation or extraction of liquids from materials. ILLINGWORTH CARBONIZATION CO., LTD., and S. R. ILLINGWORTH (B.P. 281,390, 1.9.26).—The liquid is drained on a moving porous surface, and the liquid is removed from the other side of the porous surface by other moving means such as porous belts or brushes.

B. M. VENABLES.

Salt or evaporating pans. VEREIN. SCHWEIZERISCHE RHEINSALINEN (B.P. 277,639, 25.8.27. Switz., 15.9.26).—A mechanically-driven scraper for discharging salt comprises a carriage travelling longitudinally to and fro and supporting a hollow shaft across the pan, on which scraper arms can rock freely. The hollow shaft also carries, clamped to it, shorter lifting arms. Within the hollow shaft is a solid shaft which effects the reciprocating motion of the carriage.

B. M. VENABLES.

Containers for treating corrosive liquids etc. BEACON OIL CO., and H. H. HEWETSON (B.P. 281,928, 20.4.27).—A vessel suitable for treating oils at a high pressure and at temperatures between 230° and 480° (or other liquids of which the vapours are corrosive) is constructed with an easily renewable upward extension provided with pipes for oil and vapour and with a safety valve, and the apparatus is so worked that the liquid level is always kept in the comparatively small extension, which, as a further economy, may be lined inside with corrosion-resisting material.

B. M. VENABLES.

Production of filter-sieves. A. PAHL (B.P. 281,879, 29.12.26).—A filter sieve for liquids or gases is produced by dipping a woven wire screen in molten glass or enamel, and applying small fragments of porous lime, charcoal, or chalk before the glass or enamel has time to

set. The screens when in use may be saturated with solutions of caustic alkali or chlorine.

B. M. VENABLES.

Screening of materials. J. BURR (B.P. 282,176, 22.9.26).—The screen consists of a number of longitudinal spaced bars or slots, into which fingers carried by a continuous band or chain project. The motion of the fingers is upwards as well as longitudinal so that the material is rolled over the screen, not merely pushed along.

B. M. VENABLES.

Screening apparatus. R. A. LEAHY (U.S.P. 1,650,896, 29.11.27. Appl., 8.12.23).—The screens are agitated by means of a pulsating column of liquid, which is subjected to the action of a reciprocating piston.

T. S. WHEELER.

Apparatus for effecting intimate contact of gases and liquids. S. WRIGHT (B.P. 281,958, 9.6.27).—An apparatus similar to a rectifying tower is provided with centrifugal sprayers adapted to lift liquid from each tray. The space above each tray of liquid is subdivided horizontally and the gas is caused to pass round each sub-compartment before passing to the next.

B. M. VENABLES.

Apparatus for determining the strength of solutions [by colorimetric assay]. C. KOCOUR (B.P. 270,338, 30.4.27. U.S., 1.5.26).—In addition to the vials containing the assay and standard, others are used containing colours which will neutralise the colour of the assay; e.g., sight may be taken through a green assay superposed on a red vial. Several red vials may be used containing fractions of the constituent being determined, as well as the green standards for the units.

B. M. VENABLES.

Gas detector. W. JOENS (B.P. 273,296, 21.6.27. Ger., 24.6.26).—A gas detector for indicating the presence of a constituent which differs in density from the bulk of the atmosphere to be tested comprises a porous body, an indicating device in the form of a thin metal plate gilded all over, which, when distorted by the diffusion pressure, forms an electrical contact, and a battery, the whole being contained in a stick-like container. The contact in the diaphragm chamber has a screw adjustment, and the screw may be provided with a pointer indicating the percentage of the foreign constituent in the gas.

B. M. VENABLES.

Grinding mill. J. B. C. SCHERBAUM (U.S.P. 1,656,503, 17.1.28. Appl., 11.2.27. Ger., 15.2.26).—See B.P. 266,009; B., 1927, 432.

Mixing and agitating [means for freezing] machines. C. H. CROWLEY, and WATERMOTORS, LTD. (B.P. 281,833, 30.10.26).

Pneumatic suction apparatus for the removal of dust and like finely-divided material. PNEUMATIC CONVEYANCE & EXTRACTION, LTD., and W. A. SMITH (B.P. 281,811, 5.10.26).

Dust extractors. WOODALL-DUCKHAM (1920), LTD., S. N. WELLINGTON, and A. SCOTT (B.P. 282,258, 5.4.27).

Dust separator [for gases]. A. M. GOODLOE, ASSR. to MIDWEST STEEL & SUPPLY CO., INC. (U.S.P. 1,649,220, 15.11.27. Appl., 6.12.24).

Apparatus for removing dust and other solid matters from air and other gases. D. HALL, J. H. KAY, and HALL & KAY, LTD. (B.P. 282,233, 9.2.27).

Apparatus for the removal of dust from gases. ATELIERS J. HANREZ SOC. ANON., and A. MODAVE (B.P. 277,281, 15.12.26. Belg., 13.9.26).

[Stop device for] apparatus for expressing liquid constituents from materials of various kinds. F. KRUPP GRUSONWERK A.-G. (B.P. 273,294, 20.6.27. Ger., 23.6.26. Addn. to B.P. 252,005; B., 1927, 241).

[Means for removing non-condensable gas from] refrigerating systems. W. H. CARRIER, and CARRIER ENGINEERING CORP. (B.P. 281,356, 20.7.26).

II.—FUEL; GAS; TAR; MINERAL OILS.

Lancashire coalfield. Wigan Four-feet seam. ANON. (Dept. Sci. Ind. Res., Fuel Res., 1927, Physical and Chemical Survey of the National Coal Resources, No. 10, 1927, 60 pp.).—Samples of coal taken from the Wigan Four-feet seam in ten different districts have been examined in detail. The seam is of good quality, consists of fairly hard and moderately bright coal containing a certain amount of durain, and is seldom split by thick shale or dirt bands. The thickness varies from about 2 ft. on the east to a maximum of 6 ft. 3 in. at Garswood, thence thinning slightly towards St. Helens. The seam yields a good coking coal, which is used chiefly as a house or as a gas coal; it produces a coke which is usually only slightly swollen. The moisture content is normal, about 2–3%, except for the Garswood sample (5.6%). The ash content varies from 2.6 to 9.9%, the average, however, lying between 3 and 4%. The average volatile matter content is high. The sulphur content tends to be high in the Tyldesley and Atherton districts, but elsewhere is comparatively low. The results of laboratory carbonisation assays at 600° indicate the suitability of this coal for gas manufacture. Washing tests made on three of the samples show that the coal lends itself to purification by both float-and-sink and froth-flotation methods. A. B. MANNING.

Cannel and pseudocannel coals. R. KATTWINKEL (Brennstoff-Chem., 1927, 8, 375–376).—A cannel and a pseudocannel coal from the Ruhr gave, respectively, on analysis: ash, 4.80% and 2.86%; volatile matter, 39.55% and 25.23%; carbon, 82.15% and 86.98%; hydrogen, 5.82% and 4.81%; oxygen, 5.08% and 2.80%; d 1.278 and 1.343. The results for the pseudocannel coal are characteristic of a "bright" coal, and it gives a strongly coherent coke button. A low silica content (17.1%) and high ferric oxide content (38.2%) are characteristic of the ash of the pseudocannel coal, the cannel coal ash containing 35.2% SiO_2 and 7.3% Fe_2O_3 . The latter also has a high alkali content. The difference between the two coals is maintained on low-temperature carbonisation, but they behave more similarly on high-temperature carbonisation. For the above reasons the pseudocannel coal is to be classified as intermediate between "bright" and "dull" coals, a conclusion agreeing with that based by Winter (Glückauf, 1913, 49, 1410) on a microscopical examination. W. T. K. BRAUNHOLTZ.

Spontaneous ignition of coal. D. J. W. KREULEN (Brennstoff-Chem., 1927, 6, 340–343).—Powdered coal (50-mesh), previously dried for $\frac{1}{2}$ hr. at 105°, was heated for 2 hrs. at 175° (actual temperature of the coal) in an air oven made of sheet asbestos and so designed that a thermometer dipped into the coal in the crucible and that air was constantly circulating over the sample. The partly oxidised coal was extracted with sodium hydroxide, and the quantity of humic acids produced was determined colorimetrically. This quantity increases with increasing content of volatile matter in the coal, and, in general, a definite relation exists between the two (both reckoned on the dry and ash-free coal). At the same time cases are found of coals with the same content of volatile matter having quite different susceptibilities to oxidation. For example, the Plessey and Yard seams (Newbiggin) contain 37.2% and 37.3% of volatile matter, respectively, but yield, under the above conditions, 159 mg. and 57 mg. of humic acids per g. of coal substance. By placing these two coals in adjacent crucibles in the asbestos oven, the temperature of which is gradually raised, and noting the temperature of each coal sample and of the surrounding air, the temperature of the Plessey and Yard coals is found to rise above that of the air at 115° and 162°, respectively, indicating the greater susceptibility of the former to oxidation.

W. T. K. BRAUNHOLTZ.

Reactions between oxygen and coal. W. FRANCIS and R. V. WHEELER (J.C.S., 1927, 2958–2967).—Oxidation of newly-won coal at 60° with oxygen proceeds through an adsorbed layer, which reacts with the external groupings of the ulmin molecule. These oxygenated groupings decompose giving carbon oxides and water with the formation of new, unstable oxygenated groupings. On raising the temperature in a vacuum, the latter decompose into the same products, which contain an amount of oxygen equal to that "fixed" in the oxidation. On repetition of this revivification process the decomposition products alter in amount owing to alteration in the character of the external groupings. The groupings of the soluble ulmin prepared by atmospheric oxidation of this coal at 150° are converted probably into carboxylic groups by similar oxidation in oxygen. Revivification cannot be effected to any great extent since the decomposition leaves a comparatively stable substance. G. A. C. GOUGH.

Differentiation between lignite and coal. W. FUCHS (Brennstoff-Chem., 1927, 8, 337–340).—Lignites are composed mainly of humic acids, whilst coals are almost or entirely free from them. A differentiation is thus afforded by determining the total humic acid (free and combined) present in the given sample. The free humic acid is determined by shaking the sample (10 g.) with *N*-calcium acetate (100 c.c.), filtering, and titrating the acetic acid liberated with 0.1*N*-alkali, the equivalent of humic acid being taken as 350. The humic acid combined with a metal (other than calcium) is found by making use of the permutit nature of the humates (cf. B., 1927, 833). The calcium in the original filtrate is precipitated as oxalate, and the latter titrated with permanganate in acid solution. Finally, the calcium humate is found by shaking the sample with *N*-sodium acetate and determining the calcium as above,

the reaction being the reverse of the previous one. The value found for free humic acid is probably, that for calcium humate certainly, too low, whereas the value for the humic acid combined with metals other than calcium is probably too high. The total humic acid present, however, affords a clear differentiation between typical lignites and coals, being not less than 60% for lignites (in the dry sample) and not more than 5% for coals.

W. T. K. BRAUNHOLTZ.

Alleged discovery of "caramelic acid." W. FUCHS (Z. angew. Chem., 1928, 41, 85–88).—The so-called caramelic acid which Marcusson claims to have isolated (cf. B., 1928, 76) both from humic acid and from dextrose by oxidation does not in fact exist. Material prepared from humic acid, following exactly the directions given by Marcusson, is not a definite compound, does not give a lead salt of constant lead content, and proves in fact to be partly changed starting material. The product obtained from dextrose by following Marcusson's directions is entirely different from that obtained from humic acid.

S. I. LEVY.

Reactivity of coke. I. Standardised method for the determination of comparative values. J. H. JONES, J. G. KING, and F. S. SINNATT (Dept. Sci. Ind. Res., Fuel Res., 1927, Tech. Paper No. 18, 32 pp.).—The reactivity of coke has been determined by measuring the volume of carbon monoxide produced when 100 c.c. of carbon dioxide are passed through a column of coke maintained at 950°. An apparatus suitable for such determinations is described in detail. The coke is ground to pass 10-mesh and remain on 20-mesh; the coke column, contained in a silica tube, is 7.5 cm. in length and 1.4 cm. in diameter; the rate of passage of carbon dioxide is 5 c.c./min. These arbitrarily chosen standard conditions are such that the reactivities, which are expressed as c.c. of carbon monoxide produced, will lie between 30 and 140 for most normal cokes. In general, the reactivity falls with continued passage of nitrogen or carbon dioxide over the heated coke. For each coke investigated three reactivities have been determined, (a) the initial value, (b) the constant value reached after 5 hrs. passage of nitrogen at 950°, and (c) the approximately constant value reached after continued passage of carbon dioxide at 950°. The last is not strictly constant, being further slowly reduced by prolonged passage of the gas. The same value of c is reached either directly from a or via b. Typical results for the three reactivities of good metallurgical cokes such as South Wales coke, Durham coke, and Yorkshire beehive are: (a) 69, 72, 70; (b) 69, 63, 67.5; and (c) 40, 48, 67.5, respectively. These may be contrasted with the corresponding values for horizontal-retort gas coke, by-product coke, and an active charcoal, which are: (a) 145, 138, 192; (b) 131, 112, —; and (c) 78, 95, —, respectively. The form of the curve obtained when reactivity is plotted against the volume of carbon dioxide passed over the coke appears to afford a good indication of its value for industrial purposes. There is also some evidence of a relationship between the reactivity and the hardness of a coke, the resistance to the "shatter" test increasing with decreasing reactivity. The fall in reactivity from a to b is a result of the removal

of volatile matter, but the cause of the further fall on the continued passage of carbon dioxide has not yet been fully elucidated. Some phenomena connected therewith, and in particular a transitory reactivation which occurs with certain cokes, are briefly discussed.

A. B. MANNING.

Production of carbon by the decomposition of carbon monoxide. VON WANGENHEIM (Brennstoff-Chem., 1927, 8, 385–388).—When carbon monoxide is passed over pure iron oxide at 650°, deposition of carbon occurs until the iron content of the catalyst is reduced to about 1%. The presence of hydrogen in the gas increases the carbon deposition. The iron in the carbon cannot be completely removed, either by the action of acids or by heating in a current of chlorine and volatilising the ferric chloride formed. The final carbon, containing traces of iron, was not magnetic, but was a conductor of electricity.

W. T. K. BRAUNHOLTZ.

Production of pure carbon at low temperatures. F. FISCHER and P. DILTHEX (Brennstoff-Chem., 1927, 8, 388–391; 1928, 9, 24–30; cf. preceding abstract).—Carbon is deposited when carbon monoxide or water-gas is passed over a catalyst containing ferric oxide at about 500°. The gas must be free from sulphur compounds, but even with sulphur-free gas an initial increase in the activity of the catalyst, followed by a decrease in activity, is observed. Hydrogen sulphide, produced by the action of water-gas on sulphur compounds in the iron on which the catalyst is supported, reduces the activity of the latter, but this is restored by dipping the iron in 0.1N-sodium carbonate. A soft iron, particularly free from sulphur, was found the most suitable carrier for the catalyst, which was painted on in the form of a mixture of ferric oxide and sodium silicate. The apparatus, in its final form, consisted of a vertical, externally-heated iron tube, in which U-shaped strips of iron, carrying the catalyst, were suspended. The gas passed over these strips and the deposit of carbon was knocked off them from the top of the tube and collected at the bottom, which was constricted and closed by a stopper. The composition of the gases leaving the apparatus was determined with an Orsat apparatus or, preferably, with an interferometer. Various proportions of carbon monoxide and hydrogen in the initial gas were used, a gas richer in carbon monoxide giving a decreased formation of methane (as by-product) and an increased carbon yield. The carbon was pyrophoric, and contained small quantities of iron, probably in the form of a carbide which can be dissolved with 2.5N-hydrochloric acid. Chemical and X-ray tests appear to indicate the presence of graphite in the carbon. The reactivity of the carbon towards carbon dioxide is intermediate between that of wood charcoal and metallurgical coke, removal of iron decreasing the reactivity. The sulphur-free carbon might find application in the iron industry, and carbon monoxide can be continuously regenerated from the carbon dioxide in the final gases by passing the latter over red-hot coke. As an example of the yield obtained in continuous working, the passage of 26.3 cub. m. during 173 hrs. gave 1.27 kg. of carbon without any diminution in the activity of the catalyst. The iron content of the carbon may be up to 7%, and is greater the more active the catalyst.

W. T. K. BRAUNHOLTZ.

Synthesis of methane from water-gas. F. W. HIGHTOWER and A. H. WHITE (Ind. Eng. Chem., 1928, 20, 10—15).—Further data on the catalytic reduction of carbon monoxide or dioxide to methane are provided by analysis of the gases produced when water-gas is circulated over a nickel catalyst; this was prepared on pumice and heated in an electric furnace at temperatures between 280 and 377°. The conversion into methane reaches a maximum at about 350°, but the observations are complicated by deposition of carbon, and are not proved to represent equilibrium conditions. Recirculation of the gas, after cooling to remove some of the water produced, shows that the reduction of carbon monoxide is the main reaction, but that dissociation of the monoxide to dioxide and carbon also occurs; when no water vapour is removed the reactions are modified in accordance with theoretical predictions. The reduction of carbon dioxide proceeds without any side reactions. Values of the equilibrium constants ($\log_e K_p$) for the composite reaction $\text{CO} + \text{CO}_2 + 7\text{H}_2 = 2\text{CH}_4 + 3\text{H}_2\text{O}$ have been determined and are compared with those given by calculation from data published by other workers; the new experimental results are generally lower, and suggest that equilibrium conditions have not been reached.

R. H. GRIFFITH.

Equilibrium conditions in the formation of hydrocarbons and alcohols from water-gas. H. THORSCH (Brennstoff-Chem., 1927, 8, 376).—Smith has deduced (B., 1927, 593), from calculations of the equilibrium constants, that increasing temperature should favour the formation of higher rather than lower paraffins from water-gas, whereas in reality it favours the formation of methane. The further conclusion that increased pressure should tend to produce the higher paraffins agrees with the results of experiments carried out under conditions in which these compounds remain in the catalyst. At higher temperatures, however, at which these higher paraffins are expelled from the catalyst, oxygenated substances are obtained, possibly as secondary products.

W. T. K. BRAUNHOLTZ.

Estimation of firedamp: flame caps. C. S. W. GRICE and D. W. WOODHEAD (Safety in Mines Res. Bd., Paper No. 37, 1927, 8 pp.).—The "caps" visible on the flame of a safety lamp when the latter is introduced into atmospheres containing varying amounts of firedamp have been photographed. A vignetting method was used to avoid the halation which otherwise is caused by the testing flame when sufficient exposure is given to obtain the detail of the cap. The prints were dyed to reproduce the blue colour of the flame caps, and the yellow of the testing flame was then added by hand. Coloured reproductions of these are given.

A. B. MANNING.

Blue oil of lignite producer tar and its relationship to the azulene and the sesquiterpenes of oil of camomile. S. RUHEMANN and K. LEWY (Ber., 1927, 60, 2459—2469; cf. B., 1926, 42).—Azulene is isolated from the neutral fractions of high b.p. of the tar by treatment with hydroferrocyanic acid followed by action of alkali hydroxide on the precipitate thus produced; separation of azulene from the mixture of

liberated hydrocarbons is effected by dissolution in concentrated sulphuric acid and subsequent precipitation with water. The azulene is converted into its *picrate*, $\text{C}_{15}\text{H}_{18}\text{C}_6\text{H}_2(\text{NO}_2)_3\cdot\text{OH}$, m.p. 116°. Camomile oil on treatment with hydroferrocyanic acid gives the *compound*, $\text{C}_{15}\text{H}_{18}\text{H}_4\text{FeC}_6\text{N}_6$; azulene, b.p. 163°/14 mm., derived from this source is shown to be identical with that from lignite tar by comparison of the picrates. The validity of the optical method used by Herzenberg and Ruhemann (*loc. cit.*) in the examination of azulene from lignite tar and gurjunene is thus established (cf. Ruzicka and Rudolph, A., 1926, 299). Catalytic hydrogenation of azulene in the presence of colloidal palladium gives colourless *octahydroazulene*, $\text{C}_{15}\text{H}_{26}$, b.p. 123—124.5°/10 mm., d^{15}_4 0.8967, n^{15}_D 1.4921, the optical constants of which suggest the presence of a difficultly hydrogenated double linking (cf. Ruzicka and Rudolph, *loc. cit.*) or a tricyclic system, and are not in harmony with the benzofulvene structure assigned to azulene by Kremers (A., 1923, i, 454). Further, the power to react with hydroferrocyanic acid is not exhibited by dimethyl- or diphenyl-benzofulvene or by indene or hydrindene. A fraction of camomile oil, b.p. 160—170°/14 mm., ester number 41, after being freed from azulene was hydrolysed, but the alcohol could not be isolated in substance, as hydrogen phthalate, phenylurethane, or triphenylmethyl ether; a non-crystalline benzoate was obtained. Treatment of the crude alcohol with potassium hydrogen sulphate at 180° gives a non-homogeneous sesquiterpene *A*, $\text{C}_{15}\text{H}_{24}$, b.p. 137—139°/13 mm., d^{17}_4 0.8975, n^{17}_D 1.5046. Repeated distillation of the fractions of camomile oil, of b.p. 130—140°/10 mm. and 120—130°/10 mm. yields a *sesquiterpene C*, b.p. 129—131°/10 mm., d^{20}_4 0.8998, n^{20}_D 1.5029, $[\alpha]^{20}_D + 16.13^\circ$ in chloroform, in small amount and a *sesquiterpene B*, b.p. 124—125°/10 mm., d^{20}_4 0.8689, n^{20}_D 1.4905, $[\alpha]^{20}_D - 4.35^\circ$ (*hydrochloride*, $\text{C}_{15}\text{H}_{27}\text{Cl}_2$, m.p. 45°). The compound *B* appears to be monocyclic. It is converted by hydrogen in the presence of a nickel-aluminium oxide catalyst into the *hexahydride*, $\text{C}_{15}\text{H}_{30}$, b.p. 114—115°/10 mm., d^{15}_4 0.8200, n^{15}_D 1.4531, which is not dehydrogenated to a derivative of naphthalene. Azulene does not appear to exist preformed in camomile oil, but is apparently derived by fermentative action from sesquiterpenes.

H. WREN.

Tar from Russian oil shale. G. STADNIKOV and A. WEIZMANN (Brennstoff-Chem., 1927, 8, 343—345).—Kaschper oil shale, containing 13.4% of moisture, 46.2% of ash, and 3.8% S, when distilled in a Fischer retort, gave 11.1% of tar, the yield being raised to 15% by distilling in a current of carbon dioxide. The light oil from the tar was treated with formaldehyde and naphthasulphonic acid ("Petrov's contact") to remove acidic sulphur compounds and a portion of the unsaturated compounds, and the treated oil was distilled with steam and the distillate washed with dilute sulphuric acid and dilute sodium hydroxide. The remaining oil was fractionally distilled, and the distillate boiled with and finally redistilled over metallic sodium. The fractions of the distillate (having b.p. ranging from 105° to 220°) had high sulphur contents (11.6—7.6%), and were acetylated with acetyl chloride in the presence

of stannic chloride. Analyses of the purified and re-distilled acetyl products proved the presence in the original oil of a thioxen or ethylthiophen, and of propylthiophen or one of its isomerides.

W. T. K. BRAUNHOLTZ.

Action of strong sulphuric acid on olefines and alcohols. W. R. ORMANDY and E. C. CRAVEN (J. Inst. Petrol. Tech., 1927, 13, 844—854).—The reactions involved in determining the unsaturated content of liquid hydrocarbon mixtures by absorption in sulphuric acid are investigated, attention being concentrated on oily reaction products obtained with olefines and alcohols, and sulphuric acid. Ethylene showed no hydrocarbon formation, but propylene, amylene, and mixed olefines obtained from cracked spirit all gave good yields of open-chain paraffins and highly unsaturated substances which remain dissolved in the acid. If olefines and aromatic hydrocarbons are treated simultaneously, the latter also are involved in the reaction. *iso*- and *n*-Propyl and amyl alcohols, *cyclohexanol*, and 1- and 4-methyl*cyclohexanols* similarly gave two types of reaction product, one of which remained in the acid; the oily products from open-chain alcohols were open-chain paraffins, but those from *cyclohexanols* were cyclic hydrocarbons.

R. H. GRIFFITH.

Purification of benzol for motor fuel. F. T. HATSWELL (Gas World, 1928, 88, Coking Sect., 11—14).—Crude benzol is washed with dilute acid, and, after separation of the latter, is treated with the purifying medium at 50—60° for about 1 hr. It is then filtered through flannel, neutralised, and finally distilled. The medium, which is a finely-divided mixture of an absorbent material and ferric sulphate in an active form, polymerises or interacts with the impurities and then adsorbs the products of these reactions. Large-scale tests have shown that the losses are much smaller than by the usual acid washing, and that a motor benzol is produced which, although not able to pass the acid tests, is stable, free from gumming materials and sulphur, and gives a better performance in the engine than a normal high-grade benzol. The cost of the process compares favourably with that of the usual acid washing.

A. B. MANNING.

Causes of varying sp. gr. of gas-works benzol. A. SCHNEIDER and W. KONRAD (Gas- u. Wasserfach, 1927, 70, 1188—1189).—Gas-works benzol, having *d* 0.840, was found to contain only 1.7% of paraffins. Its refractive index (1.481—1.490 for fractions of b.p. 60—83°) suggested the presence of considerable quantities of *cycloparaffins*, which would also account for the low sp. gr. These were not derived from the wash oil, but were produced in the process of gas manufacture, particularly when the carbonising temperature fell at times below 1000°.

W. T. K. BRAUNHOLTZ.

Phenols from natural petroleum. Y. TANAKA and R. KOBAYASHI (J. Fac. Eng., Tokyo Imp. Univ., 1927, 17, 127—133, and Proc. Imp. Acad., Tokyo, 1927, 3, 595—596).—The greater part of acid substances found in petroleum consists of naphthenic acids, but phenols are also present; those occurring in the kerosene fraction of Japanese petroleum have been isolated and examined. The oil is treated with strong aqueous caustic soda,

and the crude mixture obtained on acidifying this solution is redissolved in a limited amount of alkali. The difference in the degree of hydrolytic dissociation of salts of naphthenic acids and phenols makes possible the selective extraction of the latter by means of ether. The purified mixture of phenols obtained by several repetitions of this treatment was fractionated and the physical and chemical properties of the fractions were determined; *o*- and *p*-cresols and *o*- and *m*-xylenols were determined by interaction with chloroacetic acid, and *m*-cresol by formation of its trinitro-derivative. The phenols thus identified were 50% of *p*-cresol, with less *o*- and *m*-cresols, and small amounts of *m*- and *o*-xylenols, diethylphenol, and triethylphenol.

R. H. GRIFFITH.

Cholesterol as parent of petroleum. W. STEINKOPF (Ber., 1927, 60, 2609—2610).—Zelinski's view that cholesterol is the parent of petroleum (B., 1927, 865) cannot be upheld since it has not been available in sufficient quantity. It is possible that decomposition products of cholesterol may be mixed with petroleum. By use of kieselguhr in place of aluminium chloride the author has obtained from cholesterol products very similar to those described by Zelinski (cf. A., 1921, i, 24).

H. WREN.

Corrosion by oil. H. J. YOUNG (J. Inst. Petrol. Tech., 1927, 13, 760—772).—Two cases of crank-shaft corrosion in motor-ships were traced to lubricating oils which, according to standard tests, had no properties usually associated with corrosive action. A direct test has accordingly been devised in which warm oil flows continuously over slabs of steel kept at a uniform temperature; part of the surface is white-metalled with bearing metal in order to produce conditions similar to those in the engine, and the specimens are polished before treatment. Examination of numerous oils, including some contaminated with acid or other inorganic impurities, showed that determination of inorganic acid was valueless, but determination of the "sulphate value" would indicate roughly the behaviour of a lubricant; the new test is, however, more sensitive and less laborious.

R. H. GRIFFITH.

Resins in lubricating oils and their action on steel. M. W. BORODULIN (Petroleum, 1927, 23, 1515—1518).—The Akzys method is the most satisfactory for determining the content of resin-forming compounds in lubricating oils. The oil, diluted with an equal volume (or more) of standard benzine, is shaken with 95% sulphuric acid and the increase in volume of the acid layer is taken as representing the volume of the resin-forming compounds present in the oil. The modification of Armani and Rodano's method (B., 1921, 138 A) does not give satisfactory results, and may even give a negative value for the resin content. The rusting of steel plates coated with oils of varying resin content bears no relation to the latter, but depends mainly on the viscosity of the oil. Indeed, the protective action of oils on steel is enhanced by increasing their viscosity by addition of resin, and oils from which the resins have not been completely removed afford better protection than those that have been more thoroughly refined.

W. T. K. BRAUNHOLTZ.

Extension of the formolite analysis of lubricating

oils. A. M. NASTUKOV (Petroleum, 1927, 23, 1451—1452).—The Akzys test does not give the same value for lubricating oils as the fourth formolite value, although it does for crude oils. The content of saturated and unsaturated hydrocarbons, in both lubricating and crude oils, is, however, given by the formolite analysis. Further, if E is the Engler viscosity at a given temperature, F_3 the third formolite value, b the resin content, and C a constant, then $(E + b)/(F_3 + 2b - 3.5) = C$. The values of C at 100° for cylinder oils and "viscosines," and at 50° for most machine oils, were found to be 0.20, 0.22, and 0.29, respectively. The equation enables the viscosity of an oil to be calculated from its formolite value. W. T. K. BRAUNHOLTZ.

Motor spirit, with special reference to its use in internal-combustion engines. H. Y. V. JACKSON (J. Inst. Petrol. Tech., 1927, 13, 855—874).—A summary of contemporary information concerning chemical and physical properties of motor fuels, and the mechanism of combustion in the engine. R. H. GRIFFITH.

Carbon monoxide in two large garages. S. H. KATZ and H. W. FREVERT (Ind. Eng. Chem., 1928, 20, 31—36).—Continuous records of the carbon monoxide content of the air in two garages have been obtained over a period of four months, with an instrument previously described (cf. U.S.P. 1,578,666; B., 1926, 430). Each day is divided into four periods, for which maximum and average values for carbon monoxide in parts per ten thousand of air are shown. No injurious concentrations were found except for short times, or near engines running without load on a rich fuel-air mixture, but conditions might have been worse in cold weather when less efficient ventilation was possible. The behaviour of the recording instrument during long use is described. R. H. GRIFFITH.

Burkheiser ammonium sulphite-bisulphite process. TERRES and HEINSEN.—See VII.

PATENTS.

Manufacture of briquettes. L. M. JOHNSTON and J. L. FARRELL (U.S.P. 1,655,728, 10.1.28. Appl., 24.2.26).—Acid tars are used mixed with pulverised carbonaceous material, and compressed into briquettes which are then roasted to drive off the acid.

H. ROYAL-DAWSON.

Gas producer. C. W. LUMMIS, Assr. to MORGAN CONSTRUCTION Co. (U.S.P. 1,655,320, 3.1.28. Appl., 10.7.19).—A gas producer having an annular base for supporting the fuel bed is provided with a flange at the inner periphery of its base, whereby a depth of water is maintained thereon. By a mechanical device the ashes at the bottom are crowded inwardly and over the flange.

C. O. HARVEY.

Manufacture of gas from petroleum oil. E. M. CLARK, N. E. LOOMIS, and F. A. HOWARD, Assrs. to STANDARD DEVELOPMENT Co. (U.S.P. 1,651,115, 29.11.27. Appl., 27.5.20).—A heavy oil is cracked and the pitch formed is treated with steam to yield water-gas, which is carburetted with the light oil formed in the cracking and mixed with the gas similarly produced.

T. S. WHEELER.

Oxidation of hydrogen sulphide contained in industrial [illuminating] gases to sulphur. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 282,508, 27.9.26).—The gases containing hydrogen sulphide are brought into intimate contact with water or steam to give them a humidity of about 80%, and are then passed through active carbon or other highly adsorptive material which may be cooled and/or moistened to increase its humidity. W. G. CAREY.

Operation of internal-combustion engines and apparatus therefor. G. B. ELLIS. From MOORE INVENTIONS CORP. (B.P. 281,739, 14.6.26).—In an internal-combustion engine a portion of exhaust gases and/or high-pressure gases from the working stroke are injected into the inlet pipe beyond the throttle, in quantities that vary with the throttle opening, so that the fuel mixture is converted into perfectly dry gas.

B. M. VENABLES.

Retort for the distillation of oil shales. L. T. FAIRHALL (U.S.P. 1,656,107, 10.1.28. Appl., 23.4.26).—A rotatable, horizontal, cylindrical retort has two centrally and axially disposed conduits, the one extending for practically the whole length of the retort and being closed at one end, the other end extending beyond the end of the retort. The portion of this conduit situated inside the retort is perforated. The other conduit supports the closed end of the first conduit and serves for the admission of steam, which enters the retort through a number of radially disposed pipes.

C. O. HARVEY.

Oil-cracking apparatus. F. C. VAN DE WATER and F. R. SUNDERMAN, Assrs. to PETROLEUM LABORATORIES INC. (U.S.P. 1,655,030, 3.1.28. Appl., 29.8.22).—An oil still is provided with a central flue for combustion gases and with a baffle which projects inwardly from the still body and upwardly in a position substantially midway between the walls of the flue and the still body.

C. O. HARVEY.

Distillation of petroleum oil. R. W. HANNA, Assr. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 1,655,603, 10.1.28. Appl., 31.3.23).—Lubricating oils are obtained from petroleum oil fractions which would ordinarily undergo decomposition below their b.p. (under partial vacuum) by vacuum distillation under reduced pressure, while adding lighter lubricating oil fractions. The distillation is carried out at a substantially constant temperature, below that at which decomposition of the heavy oil would occur, and the vapours are withdrawn at such a rate as to cause vaporisation of both the heavy and the light (added) lubricating oil fractions.

C. O. HARVEY.

Treatment of petroleum oil. A. D. DAVID, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,655,596, 10.1.28. Appl., 14.2.23).—A furnace-heated tubular still is connected with two vaporising chambers either of which may be isolated from the system. Means are provided for introducing a cooling medium into the transfer lines communicating with the isolated vaporising chamber.

C. O. HARVEY.

Treatment of [mineral] oils. A. F. L. BELL, Assr. to ASSOCIATED OIL Co. (U.S.P. 1,655,890, 10.1.28. Appl., 11.8.24).—Oil and a treating agent are delivered

to a settling tank provided with means for the prevention of agitation. The oil is withdrawn from the top of the tank, and the treating agent may be recirculated or passed to an evaporating vessel whence the evolved vapours and unevaporated residue are withdrawn.

C. O. HARVEY.

Refining of petroleum oils. P. McMICHAEL, Assr. to HYDROCARBON REFINING PROCESS Co., INC. (U.S.P. 1,655,068—9, 3.1.28. Appl., [A] 23.10.23, [B] 14.2.24).—(A) The oil is heated and digested with an aqueous solution of a fixed alkali hydroxide (*e.g.*, with 10–33 pts. of an aqueous solution containing about 1 pt. of calcium hydroxide and 0.1 pt. of ammonium chloride per 100 pts. of oil for 1–4 hrs. at about 100°) and is subsequently treated with 70–83% sulphuric acid followed by treatment with more concentrated acid. (B) The oil is treated with a fixed alkali and ferrous hydrate in the presence of air, and subsequently with 1.5–15% of 70–83% sulphuric acid. Finally the oil is steam-distilled in the presence of a fixed alkali. C. O. HARVEY.

Refining of petroleum and oil-field emulsions. L. E. WINKLER and F. C. KOCH (U.S.P. 1,650,813, 29.11.27. Appl., 20.9.26).—A spray head is fitted below the liquid contained in a battery of tanks elevated progressively in series and connected together for the purpose of maintaining a predetermined level above the head. A gas supply pipe communicates with the spray head of the first tank, and each tank is connected, above the liquid level therein, with the spray head of the next succeeding tank. H. ROYAL-DAWSON.

Desulphurising and purifying petroleum oil. R. CROSS (U.S.P. 1,654,581, 3.1.28. Appl., 21.11.23).—The oil is purified and freed from sulphur by treatment with a sodium plumbite solution at temperatures above 93°; the solution and oil are made to flow in counter-current and the operation is carried out under sufficient pressure to prevent vaporisation of the liquid materials. C. O. HARVEY.

Refining of elaterite. R. E. HAUGHEY (U.S.P. 1,653,766, 27.12.27. Appl., 15.4.25).—The elaterite, contained in an open retort resting upon and connected (by means of a restricted opening) with an air-tight chamber, is ignited, the melted material flowing downwards through the opening into the air-tight chamber. C. O. HARVEY.

Deodorising and filtering of oils. V. C. BENJAMIN (U.S.P. 1,655,175, 3.1.28. Appl., 29.9.25).—Oil which has been heated at a high temperature with an adsorbent material is freed from the latter by passing it (along with entrained steam) through a foraminous medium contained in a closed filtering system. C. O. HARVEY.

Decolorisation of oils under pressure. P. W. PRUTZMAN, Assr. to CONTACT FILTRATION Co. (U.S.P. 1,653,735, 27.12.27. Appl., 6.12.26).—The oil, admixed with an adsorbent, is heated to a temperature above its vaporisation point under a pressure greater than that which may result from frictional resistance to movement of the oil. C. O. HARVEY.

Purification and drying of oil separated from aqueous emulsions. G. B. ELLIS. From AMER. SHEET & TIN PLATE Co. (B.P. 282,321, 13.9.26).—Oil is recovered from oil-water emulsions, *e.g.*, the emulsion

discharged from the washing machine in a typical tin-plating process described, by breaking the emulsion in a suitably designed vessel by the addition of acid and introduction of live steam, separating the oil layer as far as possible, passing it through a screen to remove large particles of foreign matter, and thence through a heated coil or some other heating arrangement wherein the water is converted into vapour. The mixture of oil and water vapour is now discharged, *e.g.*, into a stack, the water vapour being carried upward in the air draught and the oil collected and centrifuged while still hot. S. S. WOOLF.

Agent for recovery of volatile solvents [petrol]. W. RUNGE, Assr. to BREGENT CORP. OF AMERICA (U.S.P. 1,651,155, 29.11.27. Appl., 11.2.24).—Low-temperature tar distillates free from acids and phenols are employed. T. S. WHEELER.

Coke oven. J. STEPHENSON (U.S.P. 1,656,841, 17.1.28. Appl., 4.12.25. Can., 20.12.24).—See B.P. 244,772; B., 1926, 940.

Production of hard, homogeneous fuel or similar objects from peat, peat moss, lignite, etc. B. JIROTKA, Assr. to DR. O. SPRENGER PATENTWERWERTUNG JIROTKA M.B.H. (U.S.P. 1,656,859, 17.1.28. Appl., 29.5.26. Ger., 13.8.21).—See B.P. 276,471; B., 1927, 867.

Pulverising apparatus for solid fuels. G. S. LOY (U.S.P. 1,656,862, 17.1.28. Appl., 22.11.22. Fr., 6.12.21).—See B.P. 190,132; B., 1923, 917 A.

Liquid purification of fuel gases. F. W. SPERR, JUN., and D. L. JACOBSON, Assrs. to KOPPERS Co. (U.S.P. 1,656,881, 17.1.28. Appl., 7.8.24).—See B.P. 238,172; B., 1925, 872.

Refining and cracking of hydrocarbons. R. K. COLLINS, Assr. to COLLINS PROCESS INC. (U.S.P. 1,654,577—1,654,580, 3.1.28. Appl., [A] 18.5.22, [B] 23.5.22, [C] 24.3.26, [D] 28.7.26).—See B.P. 280,034 and 280,039; B., 1928, 80.

Coke conveying and quenching apparatus. SOUTH METROPOLITAN GAS Co., and C. H. SMITH (B.P. 282,861, 24.9.26).

Apparatus for separating solids (B.P. 281,479). Separation of liquids from materials (B.P. 281,390). Containers for corrosive liquids (B.P. 281,928).—See I. Lead tetraethyl (U.S.P. 1,652,812). Vanadium from petroleum soot (U.S.P. 1,651,967).—See VII.

III.—ORGANIC INTERMEDIATES.

Preparation of acetone. E. DONATH (Chem.-Ztg., 1927, 51, 924).—The author refers to his previous work (A., 1889, 230) in which he showed that acetone is formed by passing alcohol vapour over manganese dioxide (containing 90.7% MnO₂ and 2.7% BaO) heated at 540°. The bulk of the acetone is probably formed by direct oxidation, but part of the alcohol may be oxidised to acetic acid, the resulting acetates being then decomposed to acetone.

W. T. K. BRAUNHOLTZ.

Microchemical identification of lactic acid. J. GRÜSS (Woch. Brau., 1928, 45, 16–18).—A few drops

of the liquid to be tested are placed on a microscope slide and a small crystal of cobalt acetate is added. On drying, the slide is examined for characteristic reddish aggregates of cobalt lactate. If none is seen, the dry residue is quickly rinsed with a drop of distilled water which is evaporated on a clean slide, and the residue from this examined for cobalt lactate. For a further test, 3 c.c. of the liquid are distilled to half volume with 1 c.c. of strong sulphuric acid, and the distillate is collected. To a portion of the distillate a speck of sodium nitroprusside and a drop of piperidine are added, when a blue colour indicates acetaldehyde. Since this may arise from other substances than lactic acid, the remainder of the distillate is tested for the formic acid also produced, by neutralising with magnesia, boiling, and filtering. The filtrate after evaporation is tested on a slide with a speck of cerium nitrate. The characteristic rhombic dodecahedra of cerium formate may be recognised even if imperfectly formed, by the dark cross exhibited when examined between crossed Nicols. The preparation may be purified by quickly rinsing on to a fresh slide. The microscopic appearance of the crystals is figured, and details are given of results obtained in examining yeasts. F. E. DAY.

Analysis of mixtures of similar organic compounds. F. H. RHODES, F. T. GARDNER, and A. W. LEWIS (Ind. Eng. Chem., 1928, 20, 85–86).—The amount of a substance present in admixture with other isomeric, homologous, or unknown substances can be determined from the apparent average mol. wt. of the mixture in two different solvents, one of which is not a component of the mixture whilst the other is identical with the substance being determined. In the first case the true average mol. wt. is obtained, whereas in the second the amount of the particular substance present in the sample increases the amount of the solvent and a corresponding difference in the depression of the f.p. is obtained, from which the concentration of the particular component can be calculated. The method is limited to substances which have f.p. within the range of temperature covered by a Beckmann thermometer, and cannot be used when the compounds are ionised or polymerised or if they form compounds with the other components. Examples of the analysis of various synthetic mixtures of naphthalene, its nitro-derivatives, diphenyl, and a heavy coal-tar oil are described and satisfactory results are obtained.

E. H. SHARPLES.

Hydrocarbons and alcohols from water-gas. TROPSCH. **Action of sulphuric acid on olefines and alcohols.** ORMANDY and CRAVEN. **Phenols from petroleum.** TANAKA and KOBAYASHI.—See II.

PATENTS.

Manufacture of esters. H. E. BUC, Assr. to STANDARD DEVELOPMENT Co. (U.S.P. 1,651,666, 6.12.27. Appl., 22.12.22).—The reaction between the acid, *e.g.*, acetic acid, and the alcohol, *e.g.*, isobutyl alcohol, is performed in presence of a selective solvent for the ester, *e.g.*, a hydrocarbon oil. T. S. WHEELER.

Manufacture of butyric aldehyde. CONSORTIUM F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 271,103, 13.5.27. Ger., 15.5.26).—Crotonaldehyde is hydrogenated in the

liquid phase in the presence of a nickel catalyst under a pressure of 10–30 atm., at temperatures below 100°, with thorough agitation. In the liquid phase the velocity of reduction of crotonaldehyde to butaldehyde is greater than that of the latter to butyl alcohol, and the reaction is discontinued before the appearance of an appreciable quantity of alcohol. *E.g.*, 0.03 pt. of a nickel-kieselguhr catalyst (containing 15% Ni and prepared by reducing with hydrogen nickel carbonate or oxide precipitated on kieselguhr) and 1 pt. of crotonaldehyde are stirred at 85–90°, hydrogen being passed through at a pressure of 10–20 atm. After 2½ hrs. the liquid contains 74% of butaldehyde. B. FULLMAN.

Recovery of dehydrogenation products. I. G. FARBENIND. A.-G. (B.P. 262,086, 15.11.26. Ger., 27.11.25).—The hydrogen obtained in the dehydrogenation of the higher aliphatic alcohols (*e.g.*, isobutyl alcohol, by treatment with zinc oxide) may be stripped of the volatile dehydrogenation products which it contains (even after separation of the latter as usual), by washing it with the alcohol to be dehydrogenated. The dehydrogenation products may be recovered by, *e.g.*, heating the alcohol containing them. B. FULLMAN.

Extraction of guaiacol. O. MOSER, Assr. to VER. F. CHEM. IND. A.-G. (U.S.P. 1,651,617, 6.12.27. Appl., 28.12.26. Ger., 17.7.25).—Wood-tar oil is extracted with a limited amount of sodium hydroxide solution to remove substances of the guaiacol type only.

T. S. WHEELER.

Manufacture of aromatic oxamic acid halides. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 282,891, 30.9.26).—Aryloxamyl halides, *e.g.*, $\text{NIIAr} \cdot \text{CO} \cdot \text{COCl}$, are obtained by action of oxalyl halides on salts of aromatic amines (the free bases are useless for this purpose). Phenylloxamyl chloride, m.p. 82.5°, *o*-tolylloxamyl chloride, m.p. 89–90°, α -naphthylloxamyl chloride, m.p. 86°, and β -naphthylloxamyl chloride, m.p. 114–115°, are described.

C. HOLLINS.

Manufacture of anthracene derivatives and of benzanthrene. I. G. FARBENIND. A.-G., Assecs. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (B.P. 260,000, 18.10.26. Ger., 17.10.25).—Anthraquinone derivatives and benzanthrene, preferably mixed with copper or zinc dust, are sublimed and the vapours are carried in a stream of hydrogen over a hydrogenating catalyst (*e.g.*, a mixture of reduced copper and zinc) at 325–475° according to the catalyst and anthraquinone derivative used. 2-Aminoanthraquinone, sublimed with hydrogen over copper and zinc at 460–475°, gives 2-aminoanthracene, m.p. 235°. 1-Aminoanthracene, m.p. 120–130°, 2-methylaminoanthracene, m.p. 220–221°, 1-diethylaminoanthracene, m.p. 200° (from 1-diethylaminoanthraquinone, m.p. 118–120°), 2:6-dimethoxyanthracene, m.p. 255–256°, 2-methylantracene, m.p. 199°, and benzanthrene, m.p. 82–84°, are similarly obtained.

C. HOLLINS.

Manufacture of isatin derivatives and of indigoid dyes therefrom. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 282,863, 25.9.26).—Isatin α -chlorides [2-chloro-3-ketindolenines] react at 90° with aqueous sodium sulphite to give soluble products, which

may also be obtained (together with 50% of the corresponding indigo) by treating dehydroindigo bisulphite compounds (cf. Kalb, A., 1909, i, 967; B.P. 16,377 of 1909) with hot sodium carbonate or sulphite solution. 5-Chloroisatin α -chloride gives with hot sodium sulphite a yellow solution from which the new product may be salted out; it is stable to dilute acid, but hot acids convert it into dichloroindigo, and on reduction with hyposulphite it gives leuco-dichloroindigo. It gives indigoid dyes by condensation with α -naphthol and other reactive methylene-ketones. The new products contain sulphur and nitrogen in the atomic ratio 2:1.

C. HOLLINS.

Manufacture of oxidation products of acenaphthene. H. F. LEWIS, Assr. to NAT. ANILINE & CHEMICAL Co., INC. (U.S.P. 1,649,833, 22.11.27. Appl., 24.1.21).—Acenaphthene vapour mixed with air (9–12 vols.) is passed over manganese dioxide or an oxide of molybdenum or vanadium at 200–600° to yield mixtures containing according to conditions more or less of acenaphthylene, acenaphthenequinone, and naphthalic anhydride, which are fractionally condensed.

T. S. WHEELER.

Manufacture of *o*-anisidine and *o*-aminophenol ethers. J. TCHERNIAC (B.P. 282,907, 2.10.26).—*o*-Alkoxybenzamides are heated with a hypochlorite and caustic alkali solution. *o*-Methoxybenzamide gives an 80% yield of *o*-anisidine.

C. HOLLINS.

Preparation of mercaptobenzthiazoles. L. B. SEBRELL and J. TEPPERMA (B.P. 282,947, 9.11.26).—*o*-Chloronitrobenzene is treated with sodium sulphide, hydrogen sulphide, and carbon disulphide, to give 1-mercaptobenzthiazole. Alternatively, *oo'*-dinitrodiphenyl disulphide, obtained from *o*-chloronitrobenzene and sodium disulphide, is treated with hydrogen sulphide and carbon disulphide. The reaction is extended to the preparation of the 3-methyl and 3-phenyl derivatives of 1-mercaptobenzthiazole.

C. HOLLINS.

Compositions of matter suitable as emulsifying agents. I. G. FARBENIND. A.-G. (B.P. 258,551, 2.7.26. Ger., 18.9.25).—Emulsions of oils, fats, and other insoluble compounds in water are obtained, without employing an auxiliary solvent, by the help of organic sulphonic acids (e.g., Turkey red oil, isopropyl-naphthalene-sulphonic acid, ligninsulphonic acid, etc.) or their salts in conjunction with a gelatinisable substance (glue, gelatin, gum arabic, or any other substance which swells in water and gels when the warm aqueous solution is cooled).

C. HOLLINS.

Preparation of emulsifying agents. I. G. FARBENIND. A.-G. (B.P. 268,387, 28.3.27. Ger., 27.3.26. Addn. to B.P. 258,551; preceding).—An alkylated cellulose is used in place of the gelatinisable substance.

C. HOLLINS.

Manufacture of ketones. L. LEFRANC (U.S.P. 1,656,488, 17.1.28. Appl., 16.5.24. Fr., 17.5.23).—See B.P. 216,120; B., 1925, 337.

Manufacture of useful products by means of Friedel and Crafts reaction [*o*-benzoylbenzoic acid]. H. G. STONE and B. H. JACOBSON, Assrs. to E. C. KLIPSTEIN & SONS Co. (U.S.P. 1,656,575, 17.1.28. Appl., 28.12.23).—See Can. P. 254,834; B., 1926, 869.

Splitting-off sulpho-groups from anthraquinone-sulphonic acid derivatives. R. E. SCHMIDT, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,650,158, 22.11.27. Appl., 3.8.26. Austr., 8.4.26).—See B.P. 250,968; B., 1927, 743.

Manufacture of a composition of matter [halogen-substituted oxindole-3-carboxylic acids]. W. SCHOELLER and K. SCHMIDT, Assrs. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,656,239, 17.1.28. Appl., 4.3.26. Ger., 10.3.25).—See G.P. 443,099; B., 1927, 286.

Cellulose nitrate solutions (B.P. 282,172).—See V.

IV.—DYESTUFFS.

PATENTS.

Manufacture and use of new vat dyes. BRIT. DYESTUFFS CORP., LTD., and [A] S. THORNLEY, [B] A. SHEPHERDSON and S. THORNLEY (B.P. 282,852, 2.9.26. and 282,913, 8.10.26 [B]).—(A) Flavanthrone and (B) pyranthrone (and its derivatives) are treated with hydroxylamine in presence of sulphuric acid and ferrous sulphate to give (A) green and (B) brown vat dyes, which are improved by acylation. The acylated dyes become fast to chlorine after treatment with hypochlorite.

C. HOLLINS.

Manufacture of vat dyes. BRIT. DYESTUFFS CORP., LTD., W. D. ROGERS, H. EVANS, and W. F. A. ERMEN (B.P. 282,481, 13.9.26).—Pyranthrone is brominated in chlorosulphonic acid in presence of iodine. C. HOLLINS.

Manufacture of new azo dyes. I. G. FARBENIND. A.-G., Assces. of FARBENFABR. VORM. F. BAYER & Co. (B.P. 258,894, 24.9.26. Ger., 25.9.25).—Wool dyes fast to fulling, suitable also for printing on wool or silk, are obtained by coupling tetrazotised 2:2'-dichlorobenzidine or *m*-tolidine [2:2'-dimethylbenzidine] with 1 mol. of *m*-phenylenediamine-4:6-disulphonic acid and in alkaline solution with 1 mol. of 2-amino-8-naphthol-6-sulphonic acid ("γ-acid") or its *N*-substituted derivatives. With γ-acid a reddish-brown dye is produced, with *N*-aryl-γ-acids yellower shades.

C. HOLLINS.

New azo dyes and their application. BRIT. DYESTUFFS CORP., LTD., and H. W. MOSS (B.P. 282,548, 7.12.26).—Direct black shades on cotton are obtained by means of developable trisazo dyes of the type $\text{Ar} \rightarrow \text{N} \leftarrow \text{D} \rightarrow \alpha$ -naphthylamine or its 6(7)-sulphonic acid, where D is a diamine of the benzidine series, N is a 1:8-aminonaphtholsulphonic acid, and Ar is a phenylenediamine. E.g., 1:8-aminonaphthol-3:6-disulphonic acid is coupled acid with *p*-nitrodiazobenzene, then alkaline with tetrazotised benzidine or dianisidine, and the resulting diazodisazo compound is coupled acid with α -naphthylamine or its 6(or 7)-sulphonic acid; the nitro-group is reduced with sodium sulphide at any stage after the first coupling. The same result is obtained by using *p*-aminoacetanilide for the first coupling, with subsequent hydrolysis in place of reduction. The depth and fastness of the dyeings are improved by diazotisation and development on the fibre with β -naphthol, 2:4-tolylenediamine, etc.

C. HOLLINS.

Manufacture of azo dyes. I. G. FARBENIND. A.-G. (F.P. 621,209, 9.9.26).—Azo dyes containing as end-

component a dihydroxynaphthalene or a sulphonic acid thereof are treated with an arylsulphonyl halide in presence of an acid binding agent; *e.g.*, the *p*-toluenesulphonic ester of the dye *o*-anisidine \rightarrow 1:8-dihydroxynaphthalene-3:6-disulphonic acid gives on wool bluish-red shades fast to milling and light. C. HOLLINS.

Manufacture of mordant dyes. I. G. FARBENIND. A.-G. (B.P. 270,308, 27.4.27. Ger., 27.4.26).—A diazotised *o*-aminophenol or *o*-aminonaphthol is coupled with 2:6-dihydroxynaphthalene-3-carboxylic acid to give chrome olive-green to green-black wool dyes fast to washing, fulling, and potting, and suitable for single-bath chrome dyeing. Examples of suitable first components are 2-aminophenol-4-sulphonic acid, 4-chloro-2-aminophenol-6-sulphonic acid, and 4-nitro-2-aminophenol.

C. HOLLINS.

Manufacture of anthraquinone derivatives. O. Y. IMRAY. From SOC. CHEM. IND. IN BASLE (B.P. 282,853, 21.9.26).—Aminoanthraquinones are alkylated or aralkylated by heating at 100° with an aldehyde and formic acid. From 4-nitro-1-aminoanthraquinone, formic acid, and formaldehyde (or trioxymethylene), 4-nitro-1-methylaminoanthraquinone is obtained; benzaldehyde gives a benzyl derivative. The nitro-group may be reduced or may be exchanged for other groups, such as arylamino-groups. Other examples are benzylation of 1:4-diaminoanthraquinone, ethylation of 1-amino-4-anilinoanthraquinone, methylation of 1:5(8)-diaminoanthraquinone, 1-amino-4-hydroxyanthraquinone, 1- and 2-aminoanthraquinones, 1-amino-4-methoxyanthraquinone, and diamino-1:5(8)-dihydroxyanthraquinone, and the butylation (?) of 1-amino-4-hydroxyanthraquinone with crotonaldehyde and formic acid. Alternatively, either the aldehyde or the formic acid may be condensed first with the aminoanthraquinone. The products dye acetate silk.

C. HOLLINS.

Manufacture of dyes containing chromium. F. STRAUB, G. DE MONTMOLLIN, J. SPIELER, and C. VON PLANTA, ASSTS. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,656,844, 17.1.28. Appl., 1.6.25. Switz., 17.6.24).—See B.P. 235,862; B., 1926, 702.

Manufacture of hydroxyaryl-*p*-diamoanthrarufin compounds [8-diamino-2-*p*-hydroxyphenylanthrufin-6-sulphonic acids]. R. E. SCHMIDT, ASST. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,652,584, 13.12.27. Appl., 13.4.26. Ger., 17.4.25).—See B.P. 274,211; B., 1927, 743.

Manufacture of coeruleinsulphonic acids. W. HERZBERG and G. HOPPE, ASSTS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,656,483, 17.1.28. Appl., 20.4.26. Ger., 7.5.25).—See B.P. 251,968; B., 1927, 325.

Indigoid dyes (B.P. 282,863).—See III. **Carrying out exothermic reactions** (B.P. 282,559).—See X. **Diazo compounds** (B.P. 282,894).—See XXI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Measurement of the resistance of flax yarns to wear. G. F. NEW (J. Text. Inst., 1927, 18, 595—605 T).—The test samples, suspended vertically and carrying tension pieces, are submitted to rubbing through the oscillatory motion in a vertical plane of hardened steel

bars of cylindrical cross-section. The angle of contact between the yarn and the rubbing surface, and the tension in the yarn largely affect the number of oscillations withstood, and must be suitably chosen according to the material tested. Increase in fibre quality (as estimated in the trade), yarn twist, or amount of pre-spinning treatment given (within ordinary limits) produces a yarn with higher resistance to wear; boiling decreases this resistance whilst sizing greatly increases it. The results, in general, are concordant with the actual weaving behaviour of a series of yarns or sizes.

B. P. RIDGE.

Determination of oil in textiles. H. R. HIRST (J. Text. Inst., 1927, 18, 606—607 T).—A sample of the material is treated with a known volume of dry acetone for 24 hrs. at the ordinary temperature, part of the liquid withdrawn, and the total oil determined by evaporation of the solvent and weighing the dry residue. Results obtained for a number of commercial oils on wool by this method agree closely with the theoretical values, whilst oil can also be determined in the presence of soda soaps.

B. P. RIDGE.

Oxidation of cellulose in solution. I. L. KALB and F. VON FALKENHAUSEN (Ber., 1927, 60, 2514—2520).—The oxidation of cellulose by potassium permanganate in ammoniacal copper solution has been studied. With amounts of oxygen less than about 0.5 atom per $C_6H_{10}O_5$ group, the products show a continuous transition from the original precipitated cellulose to oxycellulose completely soluble in 10% sodium hydroxide. Complete solubility commences with 0.03 and 0.2 atom of oxygen in the cases of filter paper and cotton wool, respectively. Formation of products soluble in water is observed when more than 0.5 atom of oxygen is used for each $C_6H_{10}O_5$ group. The relationship between reducing power and acidity of the products derived by the use of relatively very small quantities of oxygen (whereby materials soluble in water are not formed) indicates that primary alcoholic groups of the cellulose are first converted into aldehydic and subsequently into carboxylic groups. The product obtained by the action of 2 atoms of oxygen per $C_6H_{10}O_5$ when subjected to dialysis yields 58% of residue, copper number 41.5, acid value about 240, and dissolves to a clear solution in water. From this material glycuronic acid is isolated as the cinchonine salt, m.p. 204°. The presence of this acid in the residues from the dialysis shows that it is probably contained therein in some form of chemical or adsorptive union from which it is liberated during the formation of the cinchonine salt.

H. WREN.

Fluorescence of pine bark, pine wood, sulphite pulp and liquor. O. GERNGROSS (Z. angew. Chem., 1928, 41, 50—51; cf. Gerngross, Bán, and Sándor, B., 1926, 23, 839; Gerngross, *ibid.*, 1927, 137).—A reply to the criticisms of Hägglund and Johnson (B., 1927, 371). Pine wood on heating for a short time with dilute hydrochloric acid yields a solution with a weak reddish fluorescence in ultra-violet light, whilst cotton wool dipped in this solution acquires a strong violet fluorescence similar to that shown by the aqueous extract of pine wood obtained by boiling under pressure. These observations are not in accordance with the view of Hägglund and Johnson that the fluorescence of

sulphite pulp is due to the presence of lignosulphonic acid.

W. J. POWELL.

[Fluorescence of sulphite pulp]. E. HÄGGLUND and T. JOHNSON (Z. angew. Chem., 1928, 41, 51).—A brief reply to Gerngross (cf. preceding abstract).

W. J. POWELL.

PATENTS.

Protection of wool from damage by bacteria.

I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (B.P. [A] 256,273 and Addn. B.P. [B] 261,342, 3. and 27.8.26. Ger., 3.8.25 and 10.11.25).—Alkylated naphthalenesulphonic acids are used to protect wool from attack by bacteria, but to be effective they should be applied (A) before or after (but not during) alkaline treatment of the wool, or (B) before carbonisation of the wool by acids.

D. J. NORMAN.

Washing of clothes. H. VONTOBEL (B.P. 282,588, 19.5.27).—Clothes are steeped in a cold solution of washing soda, soap, borax, and sodium perborate, to which have been added enzymes (oxidases, peroxidases, oxygenases, catalase, tyrosinase, etc.) and catalysts such as salts of the heavy metals (*e.g.*, ferrous or copper sulphate etc.), or substances containing enzyme and catalyst. The enzymes cause the perborate to give up its oxygen. After 10–12 hrs. the clothes are rubbed with soap and warm water, and rinsed in hot and then in cold water.

B. FULLMAN.

Treatment of fabrics. S. M. CADWELL and O. H. SMITH, Assrs. to MORGAN & WRIGHT (U.S.P. 1,651,751, 6.12.27. Appl., 13.3.25).—Cotton fabric employed to wrap rubber during vulcanisation is rendered more resistant to the acid developed by impregnation with mono- and di-sodium hydrogen phosphates. T. S. WHEELER.

Wetting of fibrous materials. I. G. FARBENIND. A.-G., Assees. of A.-G. F. ANILIN-FABR. (B.P. 245,098, 15.12.25. Ger., 23.12.24).—1:4-Dioxan is a satisfactory wetting-out agent for use in scouring and dyeing textile materials (cf. B.P. 275,653; B., 1927, 905).

A. J. HALL.

Production of cellulose from cellulose-containing materials.

A. CLASSEN (B.P. 279,147, 7.6.26 and 21.8.26).—Cellulose in a finely-divided form is obtained by treating cellulosic material, *e.g.*, wood meal, with concentrated hydrochloric acid, optionally in admixture with other strong mineral acids and preferably in conjunction with gaseous hydrogen chloride, at temperature below, at, or not substantially above 0°. Thus, 1 pt. of wood meal is moistened with an acid mixture containing 3.5 pts. of commercial concentrated hydrochloric acid and 1 pt. of concentrated sulphuric acid, and is then treated with gaseous hydrogen chloride in a rotary drum at 0° to –15°. When dissolution of the cellulose is complete, the ligneous matter is removed by filtration and the cellulose precipitated by the addition of suitable salts, either dry or in strong aqueous solution, *e.g.*, concentrated potassium chloride solution. The cellulose is collected, washed, and freed from the last traces of acid in a current of air or inert gas, whilst the residual acid liquor is treated with gaseous hydrogen chloride to precipitate the potassium chloride and regenerate

hydrochloric acid of sufficiently high concentration to be suitable for the treatment of a further quantity of raw material.

D. J. NORMAN.

Opening-up of materials containing cellulose.

I. G. FARBENIND. A.-G. (B.P. [A] 274,892 and Addn. B.P. [B] 276,025, 21.7. and 15.8.27. Ger., 21.7. and 13.8.26).—(A) Cellulose of high purity is obtained in good yield by exposing comminuted cellulosic material, *e.g.*, deal shavings, to the action of a current of air that has been heated at 30° and passed through nitric acid of 60% strength. (B) Alternatively, the material, *e.g.*, steamed firwood, is impregnated with warm or cold dilute (10%) nitric acid, freed from excess of acid by draining, and exposed to the action of a current of air at about 50° until a test sample is completely disintegrated when boiled with dilute sodium carbonate solution. In both cases the pulp is finally boiled with an alkaline solution.

D. J. NORMAN.

Manufacture of artificial silk and apparatus therefor. COURTAULDS, LTD., F. T. WOOD, and E. G. TURNER (B.P. 281,058, 11.10.26).—In the production of artificial filaments from cellulose esters or ethers by the dry-spinning process, uniformity of the product is ensured by providing the spinning cells with heating elements of substantially identical construction communicating in parallel with supply and discharge mains of such capacity that the flow of water is sufficiently plentiful and rapid to limit the temperature drop through the heating elements to, *e.g.*, 0.1–0.2°. Further, the spinning cells are all in communication with a common aspirating system (sufficiently large to maintain a constant rate of flow of air through the cells) through interchangeable, but not individually adjustable, outlets of the same size.

D. J. NORMAN.

Manufacture of artificial silk, bands, ribbons, etc.

W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 280,628, 16.8.26).—Cuprammonium hydroxide solutions of wood pulp, straw, or grasses give, in contradistinction to cuprammonium hydroxide solutions of cotton cellulose, products of good lustre and strength when the temperature of the spinning bath is below 30° or even below 20°, but preferably between 20° and 30°.

D. J. NORMAN.

Treatment [weighting] of artificial silks.

R. CLAVEL (B.P. 277,602, 17.11.26. Ger., 17.9.26).—The process of B.P. 266,640 (B., 1927, 295) is applied to artificial silks of all types. *Example*: Cellulose acetate fabric is passed through a bath containing 5 litres of a 10% aqueous solution of albumin and 50 g. of ammonium carbonate, and, after squeezing, is transferred to a second bath consisting of a 4% solution of tin chloride containing 10% of phosphoric acid (the latter to prevent precipitation of the tin). The dried fabric shows a greatly increased affinity for sulphonated direct dyes.

D. J. NORMAN.

Manufacture of preparations of alkylcellulose and of artificial material and articles therefrom.

I. G. FARBENIND. A.-G., Assees. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (B.P. 252,176, 12.5.26. Ger., 15.5.25).—Water-soluble cellulose ethers can be satisfactorily used in the preparation of cellulose ether compositions which neither swell nor dissolve to any

substantial extent in water if latex or a suitable quantity, *e.g.*, 2%, of a water-insoluble cellulose ether is colloidal dispersed in their aqueous solutions.

D. J. NORMAN.

Manufacture of nitrocellulose (or cellulose acetate) solutions and plastics. I. G. FARBERIND. A.-G. (B.P. [A] 251,303, and Addn. B.P. [B] 278,735 and [C] 279,771, 26.4.26. Ger., [A, B] 24.4.25).—Esters of organic acids with monoalkyl ethers of ethylene, propylene, and butylene glycols are good solvents for (A) nitrocellulose and (B) cellulose acetate and for many other compounds, including many natural and artificial resins, drying and non-drying oils, colouring agents, etc., which may with advantage be incorporated with cellulose ester lacquers and plastics. (C) The formate, acetate, and phthalate of ethylene glycol monomethyl ether are particularly suitable solvents or plasticisers (according to their b.p.) for cellulose acetate.

D. J. NORMAN.

Manufacture of cellulose nitrate solutions and plastics. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 282,172, 10.6.26).—As solvents for cellulose nitrate (alone or together with resins, colouring matters, oils, etc. of use in cellulose nitrate lacquers and plastics), there are used the oily products obtained by the catalytic hydrogenation of carbon oxides (*cf.* B.P. 227,147, 229,714, 237,030, and 238,390; B., 1925, 189, 338, 784), or fractions from these products, or the products (superior for this purpose) of their acylation, hydrogenation, or condensation. These may be mixed with one or more other solvents, *e.g.*, aliphatic alcohols or their esters, aliphatic or aromatic hydrocarbons, hydroaromatic substances, etc.

B. FULLMAN.

Viscose product. S. A. NEIDICH (U.S.P. 1,651,404, 6.12.27. Appl., 1.5.26).—Coagulated viscose is dehydrated by treatment with warm alcohol vapour to give a product with increased capacity for elongation.

T. S. WHEELER.

Recovery of sodium compounds from waste sulphite[-cellulose] liquors. C. H. MILLIGAN (U.S.P. 1,652,725, 13.12.27. Appl., 30.10.26).—The process of U.S.P. 1,545,522 (B., 1925, 707) is modified in that an organic acid, *e.g.*, stearic acid, is used in place of carbon dioxide to decompose sodium sulphide.

T. S. WHEELER.

Production of wood pulp. N. V. HANDELMAATS-CHAPPIJ "FIBRA" (B.P. 267,107, 21.2.27. Ger., 5.3.26).—The preparation of mechanical wood pulp, approximating in quality to hot-ground pulp, from logs or wood waste is described. The raw material with its natural content of moisture is suitably comminuted, freed from wood flour, sand, and other waste material, and is then dry-refined, after treatment, if necessary, with sufficient water to replace any moisture lost during the chipping operation. Provision is made for the frictional generation of heat in the dry refiner in order to promote separation of the fibre bundles by the formation of vapour therein. After dry-refining, the pulp is mixed with a suitable quantity of water and passed to a wet refiner, this operation also being conducted under conditions which cause the generation of heat by friction. The pulp is finally screened and formed into sheets. Suitable apparatus is described.

D. J. NORMAN.

Manufacture of pulp for paper production. D. R. NANJI (B.P. 280,629, 17.8.26).—Good yields of easy-bleaching pulp are obtained with a cooking time of 0.5—1 hr. at the working temperature by using a cooking liquor containing up to 4% of caustic soda at pressures approximating to 10 atm. or at temperatures corresponding to these pressures. When considerable quantities of pectins and sugars are present, the material may conveniently be pretreated with depectinising agents such as a dilute (0.5%) solution of ammonium sulphate or other neutral alkali or ammonium salt. Bulky materials such as grasses may be subjected to a preliminary acid hydrolysis with, *e.g.*, 0.5% sulphuric acid for 0.5—1 hr. at 4—6 atm., to remove non-cellulosic material. The application of this process to the treatment of bagasse from sugar cane is described.

D. J. NORMAN.

Manufacture of pulp and paper. L. BRADLEY and E. P. MCKEEFFE (U.S.P. 1,651,665, 6.12.27. Appl., 8.2.22).—A cooking liquor for the digestion of wood under pressure contains caustic soda and sodium sulphite in such proportions that the sulphite content does not exceed the caustic soda content.

D. J. NORMAN.

Preparation of stuff [size precipitant] for paper making. E. MAHLER and H. A. ROTHCHILD, Assrs. to KIMBERLY-CLARK Co. (U.S.P. 1,650,022, 22.11.27. Appl., 21.9.23).—China clay is heated with 50% sulphuric acid at 170° for 1½ hrs. to form basic aluminium sulphate solution containing suspended silica, which is used as formed to precipitate a size on the stock.

T. S. WHEELER.

Degumming of flax straw. L. N. GILLIS (B.P. 279,302, 15.2.27).—Flax straw, previously dried at a temperature not exceeding 49°, is crushed, freed from shive as far as possible, and treated, either in hank form or supported on, *e.g.*, reticulated copper trays (to preserve parallelism of the fibres), for 10—20 min. with a boiling degumming liquor made by dissolving 1.75 lb. of commercial caustic soda (90%) and 3.5 oz. of potassium dichromate in 12 gals. of water. The material is then successively washed with hot (preferably running) water, immersed in soap solution, again washed with water, and finally treated for about ½ min. in a neutralising bath containing a small quantity of sulphuric acid.

D. J. NORMAN.

Bleaching of paper [sulphate] pulp. O. KRESS, Assr. to AMER. LAKES PAPER Co. (U.S.P. 1,651,530, 6.12.27. Appl., 24.12.25).—The pulp is treated in succession with bleaching powder, sodium hydrogen sulphite, and again with bleaching powder.

T. S. WHEELER.

Manufacture of cellulose acetate. L. A. LEVY, Assr. to APEX (BRITISH) ARTIFICIAL SILK, LTD. (U.S.P. 1,652,024, 6.12.27. Appl. 2.10.26. U.K., 8.10.25).—See B.P. 240,624; B., 1926, 10.

Preparation of coconut fibres for spinning. H. WESTPHALEN (B.P. 271,900, 27.5.27. Ger., 29.5.26).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Bleaching by means of oxygen [peroxides]. G. ADOLPH and A. PIETZSCH (B.P. 268,325, 14.3.27).

Ger., 29.3.26).—The natural colouring substances present in hair and feathers are bleached by immersion in cold solutions containing hydrogen peroxide and persulphates or benzoyl peroxide, alcohol being added for the purpose of controlling the rate of bleaching; the combination of bleaching agents is more effective than hydrogen peroxide alone. [Stat. Ref. to B.P. 187,575, 180,325, and 9247 of 1913.] A. J. HALL.

Production of combined shades from sulphur dyes and ice-colours on vegetable fibre. I. G. FARBERIND. A.-G. (B.P. 266,387, 22.2.27. Ger., 22.2.26).—Cotton is padded with a hydroxynaphthoic arylamide or other coupling component for ice-colours in a bath containing also a sulphide dye dissolved as usual in sodium sulphide solution. After dyeing at 25–50° the ice-colour is developed with a diazo compound, and the dyeing is after-treated in the usual manner. A dye preparation consisting of a coupling component, a sulphide dye, and suitable additions for dissolving these may be made up. The process is useful for shading sulphide dyes towards the red. C. HOLLINS.

Dyeing, printing, or stencilling of materials made with or containing cellulose acetate. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLFIN, and W. B. MILLER (B.P. 283,081, 30.9.26).—Acetate silk is dyed, printed, etc. by any of the usual methods with nitro-derivatives of carbazoles. *E.g.*, nitrocarbazole, m.p. 164°, applied with the aid of sodium sulphuricinate, gives a greenish-yellow shade. C. HOLLINS.

Vat dyes (B.P. 282,852 and 282,913). **Azo dyes** (B.P. 282,548).—See IV.

Printing of leather (B.P. 256,195).—See XV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Technical production of sodium fluoride. A. E. J. MÜLLER (Chem.-Ztg., 1928, 52, 5–6).—Sodium carbonate is mixed with sodium fluoride mother-liquor from a previous operation and hydrofluoric acid is added with constant stirring until effervescence ceases, the temperature rising to 60–70°. The product has an acid reaction owing to the precipitation of the difficultly soluble acid sodium fluoride and also of sodium silicofluoride derived from the silica present in the fluorspar used for the preparation of the hydrofluoric acid. In order to decompose these compounds more sodium carbonate is added, until a portion of the mixture on removal gives a strong red colour with phenolphthalein, and the mixture is stirred and heated at 80–90° by passing in steam. The mother-liquor is removed from the sodium fluoride sludge as far as possible by a filter press, and the press cake of sodium fluoride containing about 50% of water is dried slowly. The final product is microcrystalline and contains 95–98% NaF.

F. R. ENNOS.

Determination of sodium borate. M. FRANÇOIS and (Mlle.) L. SEGUIN (J. Pharm. Chim., 1927, [viii], 6, 244–248).—1.91 g. of the finely-powdered borax are heated with 25 c.c. of water and 50 c.c. of glycerin until just dissolved. After cooling, 2 drops of phenolphthalein solution are added and the liquid is titrated with *N*-sodium hydroxide solution. The end-point is

sharp and, if the borax is pure, exactly 10 c.c. of *N*-sodium hydroxide solution are required. It is shown that the action of the glycerin is to transform the tetraborate into 2 mols. of sodium metaborate and 2 mols. of metaboric acid, the latter requiring 2 mols. of sodium hydroxide for neutralisation.

E. H. SHARPLES.

Burkheiser ammonium sulphite-bisulphite process. II. E. TERRES and A. HEINSEN (Gas- u. Wasserfach, 1927, 70, 1157–1161, 1193–1197, 1217–1220; cf. Terres and Hahn, B., 1927, 675).—Solubility curves for the salt pairs ammonium sulphate and sulphite and ammonium sulphate and bisulphite are plotted for temperatures between 0° and 60°. Similar curves are also drawn showing the conditions prevailing when (1) saturated solutions of ammonium sulphite in the absence of the solid phase and of a mixture of sulphate and sulphite in the presence of the solid phase are oxidised, (2) sulphur dioxide is passed into saturated solutions of a mixture of ammonium sulphate and sulphite, (3) solutions containing ammonium sulphate and bisulphite are neutralised with ammonia. In the oxidation of saturated solutions of ammonium sulphite in the absence of the solid phase, unsaturated solutions of sulphate and sulphite are formed. In the presence of an excess of solid sulphite a solid phase consisting of a mixture of sulphite and sulphate is first formed, and eventually the solid phase comprises pure sulphate. When solutions containing ammonium sulphate and sulphite are acidified, a separation of sulphate occurs at certain concentrations; in all other cases the solutions are unsaturated both as to sulphate and sulphite. Neutralisation of solutions containing sulphate and bisulphite leads first to separation of sulphite, which is later accompanied by sulphate. In practice the gases are never free from oxygen, and a mixture of ammonium sulphate and sulphite ("Burkheiser salt") in varying proportions is always obtained in the Burkheiser process.

W. T. K. BRAUNHOLTZ.

Occurrence of indium in commercial iron sulphide and its extraction. J. G. F. DRUCE (Z. angew. Chem., 1928, 41, 79).—A residue containing indium was found after continued treatment of commercial iron sulphide with sulphuric acid; indium oxide was eventually separated. The original proportion was about 6 pts. in 100,000.

S. I. LEVY.

Elimination of antimony in the refining of arsenic trioxide. C. L. READ (Ind. Eng. Chem., 1928, 20, 97–100).—The commercial preparation of white arsenic (99% As₂O₃) from arsenical flue dusts in two sublimations is not possible if much antimony is present. An investigation was carried out on the separation of the two metallic oxides from the vapours produced by washing "black dust" containing 68% As and 6% Sb. It was found that if the dust was sublimed in a tube furnace with temperature control and in a current of air a vitreous deposit containing 25–30% Sb was first formed, and later a crystalline deposit of arsenious oxide containing only 0.7% Sb. The temperature at which the vitreous deposit was best formed was 350°, and a large surface for its deposition was necessary. The current of air should be as slow as possible. The furnace temperature is immaterial. C. IRWIN.

Extraction of radium and mesothorium from radioactive chlorides in the cold. I. BASCHLOFF (Z. angew. Chem., 1928, 41, 57—59).—A description of a technical method, already patented, for the separation of radium or mesothorium chloride from barium chloride without evaporation. The mixed chlorides are dissolved in water, and a quantity of strong calcium chloride solution is added sufficient to precipitate at most one third of the barium chloride present, which then contains about twice as much active chloride as the original mixture. By repeated fractional precipitation a product rich in radium or mesothorium is obtained, together with a solution of barium and calcium chlorides, which are recovered by evaporation and crystallisation. The process is suitable for working up chloride mixtures containing as little as 0.1 pt. per million of radioactive chloride. Aluminium and ferric chlorides may also be used as precipitating agents, but as they are susceptible to hydrolysis calcium chloride is preferred.

W. J. POWELL.

Working with compressed chlorine gas in practice. R. FREUND (Chem.-Ztg., 1928, 52, 33—34).—A review of present-day practice. F. R. ENNOS.

Alteration of gas samples when kept and precautions against it. O. HACKL (Chem.-Ztg., 1929, 57, 993—994).—Samples of hydrogen and carbon dioxide, containing less than 1% of impurities, were kept in glass bottles carefully closed with rubber stoppers. After 7 weeks they were found to contain 15—20% O, together with a considerable quantity of nitrogen. In a second test, in which the rubber stoppers were dipped in paraffin after insertion, the composition of the gas was unchanged after 1 month's keeping.

W. T. K. BRAUNHOLTZ.

Production of carbon. WANGENHEIM, also FISCHER and DILTHEY.—See II. Milori blue. MÜLLER-MAGDEBURG.—See XIII. Barium compounds from starch. STERN.—See XVII. Sodium salts as germicides. LEVINE and others.—See XXIII.

PATENTS.

Apparatus for concentration of acids. O. MANTUIS (U.S.P. 1,655,019, 3.1.28. Appl., 15.8.25).—The concentrator has a closed circuitous passage through which acid flows, and is maintained at a predetermined level so that heating elements spaced apart longitudinally to the passage are totally submerged in the acid, which is heated to progressively higher temperatures in the direction of flow, vapours evolved from the acid being drawn off through a vapour outlet. W. G. CAREY.

Apparatus for the purification of impure solutions of caustic soda or the like on osmotic principles. L. CERINI (B.P. 272,211, 31.5.27. Italy, 1.6.26. Addn. to B.P. 265,126; B., 1927, 329).—The dialysing membranes are operated without forced circulation either as independent units or as units connected in parallel, so that instead of forcing counter-currents in the horizontal direction they will be established automatically in the solutions as a function of the various densities produced by the osmotic exchange.

W. G. CAREY.

Formation of sodium tungstate. W. B. STODDARD and I. HOCHSTADTER (U.S.P. 1,652,646, 13.12.27. Appl.,

30.3.22).—A mixture of a tungsten ore, charcoal, and sodium nitrate reacts autogenously on ignition to give a product containing sodium tungstate.

T. S. WHEELER.

Hypochlorite composition. M. C. TAYLOR, Assr. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,650,054, 22.11.27. Appl., 14.8.26).—The composition described in U.S.P. 1,481,003 (B., 1924, 334) is modified in that sodium fluoride is used in place of sodium carbonate.

T. S. WHEELER.

Preparation of [pure] calcium cyanide. R. W. POINDEXTER, JUN., Assr. to CALIFORNIA CYANIDE CO., INC. (U.S.P. 1,652,874 and 1,652,909, 13.12.27. Appl., [A] 5.2.27, [B] 13.4.26).—The compound of calcium cyanide and ammonia described in U.S.P. 1,596,120 (B., 1926, 946) is heated under reduced pressure at 325° until free from ammonia.

T. S. WHEELER.

Purification of the reagents used in preparing per-salts and other per-compounds. HENKEL & CIE., G.M.B.H. (B.P. 282,302, 12.7.27. Ger., 24.12.26).—A solution of the chemicals is stirred in the cold, or is boiled, with powdered silica gel, the clear decanted solution then being allowed to crystallise. The silica gel after absorbing impurities deleterious to the manufacture is regenerated by washing, treatment with acid, and heating.

W. G. CAREY.

Manufacture of caesium compounds of pure organic colouring matters. R. DELAPLACE (F.P. 621,420, 3.7.26).—A solution of caesium hydroxide is added to a solution of an acid dye, giving, especially in the case of eosin and erythrosin, solutions which absorb light of determined wave-lengths and are therefore useful in histology.

C. HOLLINS.

Recovery of lead or valuable lead compounds from lead-sulphur compounds. A. L. MOND. From NORDDEUTS. CHEM. FABR. IN HARBURG (B.P. 282,306, 8.8.27).—The lead compound is converted into lead sulphate and is then treated with concentrated sodium chloride solution at 4—5 atm. pressure at 140—150°. After settling, the liquid is passed through a filter-press and pure lead chloride crystallises therefrom.

W. G. CAREY.

Manufacture of lead tetraethyl. W. S. CALCOTT and F. L. ENGLISH, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,652,812, 13.12.27. Appl., 4.11.25).—Sodium-lead alloy is agitated at 60° in an autoclave which is connected by a vapour line with a reservoir of liquid ethyl chloride maintained at 40°, so that the pressure of ethyl chloride in the autoclave is constant.

T. S. WHEELER.

Dissolution of titaniferous materials in acids. TITAN CO. A./S. (B.P. 275,578, 2.7.27. Norw., 3.8.26).—During the dissolution of titaniferous materials, e.g., ilmenite, hydrolytic dissociation and consequent precipitation of titania is inhibited by gradually diluting the reaction mass with an aqueous solution of a salt or acid, and at the same time reducing the temperature, e.g., from 170° to 130°. By this method solutions containing 100—150 g. of TiO₂ per litre may be obtained.

C. A. KING.

Manufacture of a vanadium compound [from products of combustion of petroleum]. A. C.

REED, ASSR. to T. COTTER and E. L. LASIER (U.S.P. 1,651,967, 6.12.27. Appl., 20.2.24).—Petroleum soot containing vanadium is briquetted with sodium silicate solution and fused with sodium hydrogen sulphate to yield soluble vanadium compounds, which are extracted with water.

T. S. WHEELER.

Recovery of sulphur from ammonium polysulphide. P. KOPPE, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,656,563, 17.1.28. Appl., 7.4.27. Ger., 16.4.26).—See B.P. 269,546; B., 1928, 91.

Separation of chlorides of aluminium and potassium present in mixed solutions obtained in the treatment of leucite. G. A. BLANC (U.S.P. 1,656,769, 17.1.28. Appl., 19.10.21. Italy, 7.3.21).—See B.P. 176,770; B., 1922, 812 A.

Evaporating pans (B.P. 277,639).—See I. **Oxidation of hydrogen sulphide** (B.P. 282,508). **Cyanides** (U.S.P. 1,651,114).—See II. **Sulphite-cellulose liquors** (U.S.P. 1,652,725).—See V.

VIII.—GLASS; CERAMICS.

PATENTS.

Heat-resistant compounds. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of W. R. STEELE (B.P. 271,503, 20.5.27. U.S., 20.5.26).—Material to resist intermittent arcing etc. is made by mixing 20–40% (30%) of asbestos, 10–75% (65%) of zirconia, and 5–60% (5%) of lime; the mass is moistened, pressed into shape, and hardened by treatment with steam at 120–150 lb. pressure for 6–12 hrs. W. G. CAREY.

Manufacture of [refractory coatings for] silicon carbide articles. R. H. MARTIN, ASSR. to NORTON Co. (U.S.P. 1,653,918, 27.12.27. Appl., 18.4.25).—A protective coating impervious to kiln gases under normal conditions is formed on a refractory article containing silicon carbide by coating it with a composition of magnesia and ferric oxide and firing at a suitable temperature.

W. G. CAREY.

Annealing glass sheets and plates produced by an intermittent rolling operation. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST. GOBAIN, CHAUNY, & CIREY (B.P. 269,569, 13.4.27. Fr., 15.4.26).

IX.—BUILDING MATERIALS.

Aluminium and [its use in] cement. J. MEYER (Chem.-Ztg., 1928, 52, 4–5).—Contrary to the conclusions of Platzmann (B., 1927, 908), the author's experiments on the use of aluminium for producing porosity in the manufacture of porous concrete indicate that it is quite unsuitable. Thus, it is too expensive, and the degree of porosity obtained with any one set of materials is not constant, but is influenced by such factors as the completeness of mixing of the aluminium with the cement, and the effect of small amounts of colloidal materials in protecting the aluminium from the action of the lime solution. The crushing strength also of such porous cements containing the necessary amount of sand is much too low for them to be of technical use.

F. R. ENNOS.

Diffusion of water-soluble substances in impregnated wood. R. NOWOTNY (Z. angew. Chem., 1928,

41, 46–49; cf. B., 1926, 408).—On impregnating wood with sodium fluoride and sodium dinitrophenoxide solution, diffusion is more rapid along the outer portion of the trunk than in a direction perpendicular to the side (radial diffusion). The diffusion is followed by cutting a portion of the tree 10 months after impregnation into thin layers and examining each layer colorimetrically for the presence of either of the two substances. Fir is more suitable than Scotch pine for such experiments, since the latter is too permeable, and the zones of diffusion from the individual centres interfere with one another. Radial diffusion is slower in the layers furthest from the surface, since there is less solution available and the wood is dryer. The diffusion of both substances is very slow around the trunk; sodium fluoride, for instance, travels 2.2 cm. round the circumference in the time required for it to travel over 16 cm. along the trunk, whilst the rate of diffusion around the inner year-rings is slower than around the exterior rings. Impregnations carried out on a pole partially buried in the earth showed that rates of diffusion from the centres above and below the ground level differed considerably. Below ground, the rate in an upward direction was twice that in a downward direction; above ground the downward rate was three times the upward rate, whilst diffusion was more rapid below than above ground, owing to the higher moisture content of the wood. For preserving wood by impregnation, a network of centres of diffusion are created (by boring holes and introducing the solution), and the centres are so arranged that overlapping of the diffusion zones will take place within a short period of time.

W. J. POWELL.

Blood-albumin and its use as an adhesive for veneer and plywood. H. STADLINGER (Chem.-Ztg., 1928, 52, 8–9, 35–36).—Two commercial varieties of albumin are obtained from blood-serum albumin which is prepared from blood serum after straining off the solid material, and black albumin obtained by extracting the solid material with water. In order that the material may retain its solubility it must be carefully dried below 60° either by a hot current of air or *in vacuo*. Black albumin, which is strongly coloured by hæmoglobin, is the variety used in the woodwork industry since it is cheaper and its colour is not objectionable for this purpose. Blood-albumin is soluble in cold or lukewarm water (85–95% in commercial varieties), its solution sets by coagulation when heated at 70° or over, and the setting power is increased by the addition of electrolytes such as milk of lime or ammonia. Joints made with blood-albumin are waterproof, their strength being diminished by only 25% after soaking for several days in cold water or for several hours in boiling water. In practice, 6 pts. of blood-serum are dissolved in 11 pts. of water at 27°, 0.25 pt. of ammonia solution (*d* 0.90) and 0.13 pt. of slaked lime are added, and the solution is applied to the article, which is then placed in a press and maintained at 80–90° for a sufficient period.

F. R. ENNOS.

PATENTS.

Treating, impregnating, seasoning, and stabilising wood. G. E. RICE, ASSR. to CONSERVATION CORP. OF AMERICA (U.S.P. 1,650,738, 29.11.27. Appl., 30.8.24).

—An impregnating solution containing sucrose, maltose, and a toxic dye, *e.g.*, Crystal Violet, is employed.

T. S. WHEELER.

Impregnation of wood. D. B. BRADNER, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,652,811, 13.12.27. Appl., 19.11.25).—Wood is impregnated with a solution of arsenic pentasulphide in molten sulphur.

T. S. WHEELER.

Preservation of wood. E. B. FULKS, Assr. to AMER. CREOSOTING Co. (U.S.P. 1,652,109, 6.12.27. Appl., 9.1.26).—The wood is saturated with zinc chloride solution prior to impregnation with creosote.

T. S. WHEELER.

Fibrous paint [for coating walls]. G. E. HEYL (U.S.P. 1,656,198, 17.1.28. Appl., 29.6.26. U.K., 17.3.26).—See B.P. 259,826; B., 1926, 1016.

Wood-preserving composition. K. H. WOLMAN (U.S.P. 1,656,804, 17.1.28. Appl., 9.8.24. Austr., 23.5.24).—See B.P. 229,179; B., 1925, 284.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Energy losses in arc furnaces for steel. S. KRIZ (Arch. Eisenhüttenw., 1927, 1, 413—419; Stahl u. Eisen, 1928, 48, 71—72).—The losses of electrical energy during the melting of the charge in an arc furnace making steel are distributed as follows: transformer 3%, leads 6%, cooling water in electrode holders 4%, radiation and conduction from the walls of the furnace 15%, radiation from openings in the furnace 7%, and loss of heat in escaping gases 4%; thus 61% of the energy input is utilised in melting the charge. The corresponding loss figures for the refining of the molten charge are 6%, 3%, 7%, 29%, 11%, 7%; in this operation, therefore, only 37% of the energy input is usefully employed.

A. R. POWELL.

Rapid determination of sulphur in pig iron and steel. J. CIOCHINA (Z. anal. Chem., 1927, 72, 301—303).—A sample of the iron (1 g.) or steel (2 g.) is heated in a current of dry hydrogen at a temperature of 1240—1320° in an electric furnace. The sulphur is quantitatively converted into hydrogen sulphide, which is absorbed in a solution containing sodium, cadmium, and zinc acetates, and determined by titration with iodine and thiosulphate. The hydrogen should be washed by sodium carbonate and lead acetate solutions, and carbon dioxide should be led through the furnace at the beginning and at the end of each determination. The results obtained are usually higher than those given by dissolving the metal in dilute acid and determining the hydrogen sulphide formed, whilst the time taken for each determination is considerably less.

F. S. HAWKINS.

Alloys resistant to hydrochloric acid. B. WAESER (Chem. Fabr., 1928, 17—18).—A review of recent work, together with a table showing the composition and resistance to attack by hydrochloric acid solution of various strengths of 39 American acid-resisting alloys, 3 German special steels, and 2 ferrosilicon alloys.

A. R. POWELL.

Utilisation and behaviour of zinciferous [iron] ores, especially Meggen burnt pyrites, in the blast furnace. M. PASCHKE (Arch. Eisenhüttenw., 1927, 1,

387—402; Stahl u. Eisen, 1928, 48, 102).—Burnt pyrites containing zinc and sulphur may be smelted directly in iron blast furnaces provided that it is converted into porous briquettes, *e.g.*, by sintering in rotating kilns or in Dwight-Lloyd furnaces followed by grinding and briquetting. The sinter from either furnace should not be charged directly to the blast furnace as it requires a high temperature for reduction and entails a high coke consumption as well as giving a low-grade zinc fume. The addition of sodium chloride to the briquettes results in a higher recovery of zinc (87%) and a better quality of iron. The sintering operation may be avoided by adding the burnt pyrites directly to liquid ferromanganese slag together with coke dust, and blowing the mixture with hot air, whereby both sulphur and zinc are volatilised and a synthetic iron ore suitable for reduction in the blast furnace is obtained.

A. R. POWELL.

Extraction of zinc and other metals from ores. H. E. COLEY (Bull. Inst. Min. Met., 1928, [280], 11 pp.).—The theoretical view taken is that carbon in a "volatile" or nascent condition is a more active reducing agent than carbon monoxide, which reacts only at comparatively high temperatures. The active form of carbon is formed at the point of contact with heated ore by dissociation of hydrocarbons which have been kept cool and undissociated up to this point. In treating zinc ores, the ore, ground to $\frac{1}{4}$ -mesh, is preheated to about 500°, fed through a seal to a rotary furnace maintained at this temperature, and, by means of baffles, given contact with dissociation products of hydrocarbons injected into the tube furnace. The time for reduction is about 3 hrs., and the residue is removed through a seal excluding air, the zinc vapours passing in contra-direction to the ore. The temperature required is 300—350° lower than that of present practice, and a recovery of 97.3% has been obtained on commercial-scale working, which presented no difficulties of a chemical nature. The practical difficulties arising are of a mechanical nature associated with the thermal expansion of the furnace and the maintenance of air seals at high temperature.

C. A. KING.

Tin plate and the electrochemical series. E. F. KOHMAN and N. H. SANBORN (Ind. Eng. Chem., 1928, 20, 76—79).—Experience shows that many of the phenomena of the corrosion of tin cans containing fruit are not such as are capable of simple explanation by a tin-iron galvanic couple with tin as cathode. Corrosion tests were carried out with specimens of base plate and sheet tin immersed in fruit juice in sealed glass bottles. Corrosion was determined by weighing, and electrical potentials were also determined. It was found that the corrosion of iron was greater than that of tin when the specimens were not in contact. When they were in contact, that of iron was decreased and that of tin increased so that the relative positions of the metals in the electrochemical series were reversed. When the area of iron relative to tin was increased the protection of the former was decreased. Iron was usually anodic to tin at the beginning of the test, but then became cathodic. These results may be explained by polarisation or differences in hydrogen overvoltages.

C. IRWIN.

Thermal expansion of beryllium and aluminium-beryllium alloys. P. HEDNERT and W. T. SWEENEY (U.S. Bur. Standards, Sci. Paper No. 565, 1927, 22, 533—545).—The coefficient of expansion of beryllium (98.9%) increases rapidly with temperature, the average values per °C. for temperature intervals being 12.3 (20—100°), 13.3 (20—200°), 14.0 (20—300°), 14.8 (20—400°), and 15.5 (20—500°), all $\times 10^{-6}$. Beryllium expands considerably less than the other elements of its sub-group. Expansion of aluminium-beryllium alloys decreases with increase in the content of beryllium and increases with temperature, being also greater on a second heating of the alloy. An alloy containing 30% Be has coefficients of expansion approximately 20% less than those of aluminium. Values for a range of alloys containing 4—33% Be are given.

C. A. KING.

Alloys of beryllium with copper, nickel, cobalt, and iron. G. MASING (Z. Metallk., 1928, 20, 19—21).—Conductivity measurements show that the saturated solid solution of beryllium in copper contains 2.4% Be at 850°, and only 0.75% Be at 400°, hence alloys containing 0.75—2.4% Be undergo age-hardening after quenching from 850°. The hardness of the quenched alloys increases from 65 to 100 with increase of beryllium within the above range, and that of the corresponding alloys after age-hardening at 300—350° from 70 to 440. The elastic limit, yield point, tensile strength, elongation, and bending strength of the 2% Be alloy quenched from 800° are, respectively, 6.2, 11.0, 51.3 kg./mm.², 46%, and 45 kg./mm.²; the corresponding values after ageing at 350° are 25, 74, 87 kg./mm.², 4%, and 163.7 kg./mm.². The electrical conductivity of copper is considerably reduced by addition of beryllium, but the resistance to corrosion is increased. Alloys containing 5—10% Be quenched from above 600° consist entirely of the hard β -solid solution, and cannot be worked cold; below 590° the β -alloy decomposes into $\alpha + \gamma$, when the alloys may be extruded satisfactorily, but not cold-worked. Beryllium dissolves to a limited extent (about 2%) in nickel, cobalt, and iron, and as the solid solubility is greater at 1100° than at 600° the alloys may be heat-treated and aged in a similar manner to the copper alloys.

A. R. POWELL.

Age-hardening phenomena in beryllium-copper alloys. O. DAHL (Z. Metallk., 1928, 20, 22—24; cf. preceding abstract).—The maximum hardness (440) of the copper alloy containing 2.5% Be is obtained by ageing for 1—4 hrs. at 300—350°; at lower temperatures the hardness rises only slowly with the time of ageing, 16 hrs. being required at 250° to obtain a hardness of 320°. At 450° the finely-dispersed β -particles agglomerate rapidly, and a maximum hardness of 180 is obtained in 45 min., further heating resulting in a rapid fall in the hardness number. The optimum quenching temperature is about 840°; at lower temperatures the time required to obtain the maximum hardness is increased, and the value obtained is lower. During ageing at 150—250° the electrical conductivity first falls slightly, then rises at a rate which is greater the higher the ageing temperature; this behaviour is similar to that of duralumin under the same conditions, and appears to indicate that there are two reactions

proceeding during the ageing of beryllium-copper alloys. Microscopical and röntgenographic investigation has so far failed to establish the nature of these reactions.

A. R. POWELL.

Corrosion research. J. CZOCHRAŁSKI and E. SCHMID (Z. Metallk., 1928, 20, 1—7).—The rate of corrosion of a metal in wire or strip form may be followed by determining the tensile strength and ductility (elongation) after immersion for varying periods in the corroding medium, or by immersing the specimen under tension in the medium and observing the time that elapses before breaking occurs. Tests carried out in this manner with aluminium showed that hard-drawn wire dissolves more readily than annealed wire, whereas the converse is true in the case of copper. The dezincification of ($\alpha + \beta$)-brass in 17% hydrochloric acid at 50° is confined entirely to the β -constituent, which may be dissolved practically completely from a 3 mm. wire before any attack on the α -constituent is apparent; if the brass contains lead this also dissolves. Thus, a wire consisting of 57.9% Cu, 40.4% Zn, and 1.7% Pb was freed from the β -constituent after immersion for 208 hrs. in the acid, and then assayed 72% Cu, 27.9% Zn, and 0.05% Pb.

A. R. POWELL.

Gases in metals. III. Determination of nitrogen in metals by fusion in vacuum. L. JORDAN and J. R. ECKMAN (U.S. Bur. Stand., Sci. Paper No. 563, 1927, 22, 467—485).—For the determination of nitrogen in metals, the metal is fused *in vacuo* in a high-frequency furnace and the gases are absorbed by calcium vapour contained in an iron tube enclosed in a silica combustion tube heated at about 800°, the whole system being evacuated; this method should determine also uncombined nitrogen in a metal. The resultant calcium nitride is decomposed by hydrogen chloride and ammonia determined in the usual manner. Similar values for nitrogen were obtained by the acid-dissolution and vacuum-fusion methods in the analyses of aluminium, zirconium, and chromium nitrides, and appreciably higher values by the latter method for nitrides of silicon, titanium, and vanadium, and also for high-silicon ferrous alloys.

C. A. KING.

Cohesion at soldered surfaces. T. B. CROW (Trans. Faraday Soc., 1927, 24, 159—161).—A number of tensile tests on soldered joints are described from which an attempt is made to relate the tensile strength of the joint to that of the solder.

R. W. LUNT.

Corrosion by oil. YOUNG. Lubricating oils and steel. BORODULIN.—See II. Aluminium and cement. MEYER.—See IX. Precipitation of dust. DEUTSCH.—See XI.

PATENTS.

Cupola furnace. O. WEICHEL (B.P. 272,216, 1.7.27. Ger., 2.6.26).—In a cupola furnace provided with more than one row of nozzles, the doors of the nozzles in a vertical line are carried on one spindle, constructed with hollow and solid members so that each carries a separate door. The whole is enclosed by the air box.

C. A. KING.

Bright-annealing furnace. SIEMENS-SCHUCKERT-WERKE G.M.B.H., Assees. of T. STASSINET (B.P. 262,803, 9.12.26. Ger., 12.12.25).—Vapours produced in annealing

pots when bright annealing are removed with a slow current of inert gas by maintaining the entire length of the gas outlet above the b.p. of the vapour constituents, either by heat insulation or by electrical heating coils.

C. A. KING.

Manufacture of steel. J. K. SMITH, Assr. to GRANULAR IRON CO. (U.S.P. 1,651,638, 6.12.27. Appl., 10.1.25).—When iron produced directly in the solid form from the ore is used in the open-hearth furnace, the charge is covered with a layer of molten pig iron to inhibit oxidation.

T. S. WHEELER.

Treatment of ferrous metal [steel]. W. F. HODGES, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,651,891, 6.12.27. Appl., 26.4.24).—Decarbonisation of steel by hydrogen during brazing is inhibited by previously plating it with copper.

T. S. WHEELER.

Elimination of phosphorus from pig iron. R. SCHENCK, Assr. to VEREIN. STAHLWERKE A.-G. (U.S.P. 1,650,157, 22.11.27. Appl., 24.12.25. Ger., 11.9.24).—The metal is blown with carbon monoxide in presence of calcium oxide to convert phosphorus into calcium phosphate.

T. S. WHEELER.

Heat-resisting metallic articles. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of W. E. RUDER (B.P. 258,295, 14.9.26. U.S., 14.9.25).—Articles made from a malleable alloy of iron and chromium, containing at least 10–30% Cr, are subjected to heat-treatment in the presence of aluminium, whereby the latter is caused to alloy with the surface of the article so as to form a protective layer. The aluminium may be applied either by placing the article, surrounded with a mixture of aluminium, ammonium chloride, and zinc, into a rotatory oven, heating at about 450°, and then firing at 700–800°; or, in the case of wires, by passing them through a bath of molten aluminium and then firing; or the article may be sprayed with a mixture of powdered aluminium and a suitable liquid binder and then fired at 750–900°.

M. E. NOTTAGE.

Joining of metals. F. C. KELLEY, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,655,273, 3.1.28. Appl., 13.4.23).—The ferrous metal article is coated with a film of molten copper in the presence of a reducing agent, and a copper article may then be brazed on to the coated surface.

M. E. NOTTAGE.

Production of aluminium conducting material of high electric conductivity, strength, and chemical resistance. T. GOLDSCHMIDT A.-G. (B.P. 269,580, 14.4.27. Ger., 19.4.26).—The material, *e.g.*, aluminium alloy, after glowing, quenching, and, if necessary, cold compression, is stored at room temperature for 3–5 days, and is subsequently heated at about 200° for 10–20 hrs. [Stat. ref. to B.P. 260,275.]

M. E. NOTTAGE.

Extraction of platinum from ores etc. J. W. MELLOR (B.P. 282,543, 18.11.26).—Finely-divided platinumiferous ore is agitated with, or blown by means of a current of producer gas into, a mass of molten lead or other metal which will alloy with the platinum, and the gangue is subsequently blown off the surface of the metal. The process is repeated until the molten metal is sufficiently rich in platinum for further treatment, *e.g.*, cupellation in the case of lead. The addition

of a small quantity of alkali or alkaline-earth metal to the lead improves its power of removing platinum from the ore.

A. R. POWELL.

Metal alloys. G. C. DEETER (U.S.P. 1,656,695–6, 17.1.28. Appl., [A, B] 20.4.26).—Alloys for scavenging purposes consist of (A) 40–50% Ni, 3–10% Sn, 0.3–1% P, 1.5–5% Mn, and the balance copper, or (B) 5–10% Ni, 3–10% Sn, 0.75–5% Mn, 0.25–3% P, and 72–92% Cu.

F. G. CROSSE.

[Amalgams for] carrying out exothermic chemical reactions. E. C. R. MARKS. From SELDEN CO. (B.P. 282,559, 10.1.27).—The temperature of exothermic reactions (*e.g.*, air oxidation of naphthalene to phthalic anhydride, or anthracene to anthraquinone in presence of catalysts) is controlled by a jacket containing an alloy or amalgam of metals one of which boils below and one above the desired reaction temperature. *E.g.*, a cadmium amalgam containing 12% Cd (b.p. 778°) and 88% Hg (b.p. 357°) is semi-liquid at 20° and has b.p. 370°; the amalgam with 40% Cd is liquid at 151° and boils at 430°; with 25% Cd, m.p. 100°, b.p. 400°. An alloy of 10% Sn, 10% Pb, and 80% Hg has m.p. 20°, b.p. 370°; of 40% Sn, 40% Pb, and 20% Hg, m.p. 160°, b.p. 450°; of 30% Sn, 30% Pb, and 40% Hg, m.p. 120°, b.p. 405°. The amalgams have higher heat conductivity and lower density than mercury alone.

C. HOLLINS.

Production of protective and resistant coatings on metals. SIEMENS & HALSKE A.-G., Assees. of C. G. FINK and L. C. PAN (B.P. 263,183, 17.12.26. U.S., 18.12.25).—After plating a foundation metal with a protective metal, *e.g.*, chromium, the plated metal is immersed in molten lead to eliminate "pin-hole" defects. Lead adhering to the plating is removed by abrasion or acids.

C. A. KING.

[Anti-corrosive] treatment of metals. G. H. HOWE, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,655,269, 3.1.28. Appl., 1.4.25).—Metallic articles are protected from oxidation by covering them at ordinary temperatures with a coating composed of aluminium powder and a liquid nitrocellulose binder which wets and firmly adheres to metals at ordinary temperatures and volatilises at higher temperatures. The coating is dried and then fired at a temperature at which the aluminium alloys with the metal and above that at which the binder volatilises.

M. E. NOTTAGE.

Coating articles of various kinds with metal. E. D. FELDMAN (B.P. 261,017, 5.11.26. U.S., 6.11.25).—Articles made from non-conducting materials such as wood, felt, tile, etc. are coated with a sticky substance, *e.g.*, varnish or bitumen, on to which an alloy of lead and tin is sprayed. The article is then electroplated with the desired metal.

C. A. KING.

Thermostatic material. P. H. BRACE, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,650,979, 29.11.27. Appl., 12.3.25).—A thermostatic strip suitable for use at 800° comprises elements of molybdenum and of an iron alloy containing 22% Co and 22% Cr, both elements being chromium-plated.

T. S. WHEELER.

Pyrometer thermocouple [for immersion in molten metals]. O. BROPHY (U.S.P. 1,651,750, 6.12.27.

Appl., 27.3.23).—The elements comprise a central core and a sheath, the latter being plated with a resistant metal, *e.g.*, tungsten. T. S. WHEELER.

Concentration of mineral ores in sluice boxes etc. F. H. NASH (B.P. 282,298, 21.6.27).—A sluice box for treating mineral ores is provided with an agitator substantially of the form of an agricultural harrow which is capable of vertical movement and also of a horizontal reciprocating motion so as to disturb the bed to any desired depth. C. A. KING.

Concentration of ores and minerals by flotation. [A] C. C. SMITH and [B] W. A. DOUGLAS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,652,060 and 1,652,099, 6.12.27. Appl., [A] 10.1.27, [B] 25.10.26).—(A) Mercaptobenzthiazole, mixed with sodium sulphite, produces selective flotation of lead sulphide in presence of zinc sulphide. The latter is recovered when further quantities of the thiazole and copper sulphate are added. (B) An alkyl xanthoformate is used. T. S. WHEELER.

Deoxidiser. C. J. RODMAN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,652,496, 13.12.27. Appl., 21.12.23).—A composition for treating the atmosphere of transformers comprises a finely-divided metal, lower in the electrochemical series than hydrogen, capable of combining with oxygen at ordinary temperatures but not capable of decomposing water, *e.g.*, copper, and a catalyst for the oxidation, *e.g.*, calcium chloride. T. S. WHEELER.

Desulphurising of molten metal and briquettes used therefor. R. WALTER (U.S.P. 1,651,492, 6.12.27. Appl., 16.3.22. Ger., 8.4.21).—See B.P. 179,146; B., 1923, 783 A.

Alloy suitable for loading telephone and telegraph conductors. W. S. SMITH and H. J. GARNETT (U.S.P. 1,656,764, 17.1.28. Appl., 29.4.26. U.K., 10.7.25).—See B.P. 261,060; B., 1927, 80.

XI.—ELECTROTECHNICS.

Electrical precipitation of metalliferous dust from industrial gases. W. DEUTSCH (Z. Metallk., 1928, 20, 25—27).—A brief description with diagram of the Lurgi modification of the Cottrell electrical gas-purification plant and a review of its uses in the recovery of lead, zinc, silver, and alumina from metallurgical gases. A. R. POWELL.

Inexpensive cell for the purification of colloids by electrodialysis. R. BRADFIELD (Ind. Eng. Chem., 1928, 20, 79—80).—The cell is constructed from a rubber storage battery case sawn into three. The edges of each section are squared so that water-tight joints can be made by merely tightening up the brass bracing rods which hold the whole together, and these joints hold the parchment or other membrane. The cathode is of nickel or copper gauze and the anode of platinum or carbon, the electrodes being similar in area to the membranes. With parchment the anion requires longer time for removal than the cation. C. IRWIN.

Tin plate. KOHMAN and SANBORN. **Conductivity of aluminium.** BOHNER.—See X. **Quinhydrone and hydrogen electrodes.** SNYDER.—See XVI.

PATENTS.

[Electric] induction furnace. P. H. BRACE, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,655,983, 10.1.28. Appl., 2.4.27).—A crucible containing a metallic charge is arranged within an induction coil, and a partial core of magnetic material projects partially into the crucible so that one end of the core is surrounded by the metallic charge. J. S. G. THOMAS.

Insulation of finely-divided magnetic material [for dust cores]. H. H. LOWRY, Assr. to BELL TELEPHONE LABORATORIES, INC. (U.S.P. 1,651,957—8, 6.12.27. Appl., 3.1.27; cf. U.S.P. 1,647,737—8; B., 1928, 59).—The particles of the nickel-iron alloy used are coated with a mixture (A) of magnesium borate and silica or (B) of zinc and magnesium oxides.

T. S. WHEELER.

Insulated electrical conductor. F. L. ROMAN and H. T. WINSEMIUS, Assrs. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,649,924, 22.11.27. Appl., 12.12.25).—A fireproof coating for wire comprises cotton impregnated with a mixture of crude chloronaphthalenes.

T. S. WHEELER.

Manufacture of [gas-filled] incandescence electric lamps. D. S. GUSTIN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,651,387, 6.12.27. Appl., 22.6.23).—The lamp is filled with a mixture of argon and hydrogen (10%) containing a small quantity of the vapour of a carbon compound free from oxygen and oxidising material, *e.g.*, *p*-dibromobenzene or aniline.

T. S. WHEELER.

Electric incandescence lamp. H. WOLFF, F. KOREF, and F. SKAUPY, Assrs. to GEN. ELECTRIC Co. (U.S.P. 1,655,488, 10.1.28. Appl., 3.11.25. Ger., 18.3.25).—An electric lamp contains a gaseous filling, *e.g.*, hydrogen, including a substantial amount of halogen acid gas, at a sufficiently high pressure to reduce evaporation of the material operated at incandescence.

J. S. G. THOMAS.

Incandescence electric lamp. G. HOLST, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,655,502, 10.1.28. Appl., 11.3.27. Holl., 10.4.26).—A filament is secured to leading-in wires extending into a sealed envelope, and an auxiliary electrode, provided with a material which in response to an electrical discharge evolves a gas forming with the vaporised material of the filament a compound which is more transparent than the latter, is connected so as to act as an anode to a portion of the filament.

J. S. G. THOMAS.

Production of highly emitting oxide cathodes for electron tubes. E. SCHRACK and R. ETENREICH (RADIOWERK E. SCHRACK) (B.P. 275,542, 18.11.26. Austr., 6.8.26).—Material containing an alkaline earth is applied to a carrier material by means of a spray producing a finely-divided mist or by sprinkling. *E.g.*, a tungsten wire is coated with a thin layer of carbon by annealing the wire in an atmosphere of hydrogen saturated with tetrahydronaphthalene or containing acetylene, and sprinkled with a suspension of strontium carbonate in dilute alcoholic resin solution. The strontium carbonate is reduced to oxide by heating at 800° *in vacuo*.

J. S. G. THOMAS.

Manufacture of cathodes for thermionic valves. GEN. ELECTRIC CO., LTD. From A. JUST (B.P. 282,539, 10.11.26).—A refractory metal, *e.g.*, molybdenum, is mixed or coated with about 3% of a mixture of an alkaline-earth oxide with a very small proportion of either another alkaline-earth oxide or with an oxide of the rare-earth metals, *e.g.*, thorium oxide or praseodymium oxide, the mixture being made up in the approximate ratio of 1000 : 1. J. S. G. THOMAS.

Vacuum tube [thermionic valve]. L. WINKELMANN (U.S.P. 1,650,921, 29.11.27. Appl., 9.5.23).—The performance of thermionic valves is improved by coating the electrodes or the inner surface of the bulb with a mixture of phosphorus and a radioactive substance. T. S. WHEELER.

Storage battery. T. A. EDISON, ASSR. to T. A. EDISON, INC. (U.S.P. 1,651,196, 29.11.27. Appl., 14.5.23).—Separators of suitable porosity for use in cells of the nickel oxide-iron type are formed of asbestos fibre mixed with salicylic acid, which is afterwards removed by extraction of the material with potassium hydroxide solution. T. S. WHEELER.

Electric furnace for melting or producing glass, water-glass cement, or other substances. C. E. CORNELIUS (U.S.P. 1,656,510, 17.1.28. Appl., 26.2.26. Swed., 23.3.25).—See B.P. 249,554; B., 1927, 303.

Gas detector (B.P. 273,296).—See I. **Heat-resistant compounds (B.P. 271,503).**—See VIII. **Aluminium conductors (B.P. 269,580).**—See X.

XII.—FATS; OILS; WAXES.

Detection of coconut oil in cacao butter and cocoa preparations. F. HÄRTEL (Pharm. Zentr., 1928, 69, 37—41).—The test is based on repeated determinations of the Reichert-Meissl and Polenske values, coconut oil being richer in the volatile fatty acids than cacao butter. The first determination does not give the value corresponding to the full content of volatile acids; after a first determination, the residue is treated with a further definite quantity of water and the distillation continued; after this second treatment, a third is carried out in the same way. From the series of values so obtained, adulteration of the cacao butter may be detected and roughly estimated. S. I. LEVY.

[Extraction of oil from] oil palm in Malaya. B. BUNTING, B. J. EATON, and C. D. V. GEORGI (Malayan Agric. J., 1927, 15, 353—371).—The preparation of a palm oil of low acidity necessitates modification of the native processes of West Africa; the essential features of the process adopted in Malaya are: (a) use of only ripe, undamaged fruit, (b) sterilisation of the fruit before treatment in order to destroy the naturally occurring enzymes which promote development of free fatty acidity, and (c) rapid extraction of the oil. The sterilisation is effected by heating the fruit under slight steam pressure, after which it is transferred to a digester consisting of a vertically arranged cylindrical vessel fitted with beaters whereby disintegration of the oil cells is effected. It is then transferred to a centrifugal extractor containing a perforated basket and having a well-fitting lid provided with a jointed steam pipe whereby steam is injected into

the mash while the centrifuge is being operated, thereby facilitating the expulsion of the oil. The period of oil extraction is 15 min. and the oil is purified and freed from sludge and moisture by centrifuging in a machine of the De Laval type. Between 85 and 87% of oil in the pericarp can be recovered efficiently, and the acidity of the oil is about 3% (as palmitic acid).

H. M. LANGTON.

Atmospheric oxidation of methyl and glycol esters of β -eleostearic acid. A. B. MILLER and E. CLAXTON (Ind. Eng. Chem., 1927, 20, 43—48).—The physical and chemical changes occurring during controlled air-oxidation of β -eleostearic acid and its methyl, glycol, and glycerol esters have been examined. After 63 hrs. at 82° the methyl ester absorbed 13% O and was still fluid and oily; the glycol ester after 45 hrs. absorbed 11% O and was a sticky, elastic gel; the acid after 29 hrs. absorbed 6.5% O and formed a tough, elastic gel; and the glycerol ester formed a short, friable gel and absorbed only 3% O during 19 hrs. heating. In all cases there was an increase in hydroxy-acid content, and the results indicated the presence of polymerides containing considerable amounts of oxygen and condensation products of β -eleostearic acid. Evidence of ketol and enol formation was also noted. It is suggested that an important function of the alcohol valency is in determining the stability of the ester and preventing the liberation of free acid which, by condensation, actively influences gel formation. E. H. SHARPLES.

Phytosteryl acetate test and the phytosterol of sunflower seed oil. J. ALLAN and C. W. MOORE (J.S.C.I., 1927, 46, 433—434 T).—This test, whilst still of use in the detection of vegetable in animal fats provided that *isocholesterol* and *bombicsterol* are known to be absent, is not suitable for the detection of animal in vegetable fats since phytosterols which yield acetates melting below 125° have been isolated (cf. Power and Browning, B., 1914, 885; Steuart, *ibid.*, 1923, 462 A). It is now shown that the *phytosterol* $C_{29}H_{49}OH$, which occurs in sunflower seed oil in normal amount and constitutes the major portion of the phytosterols present yields an *acetyl* derivative, m.p. 119—119.5° (corr.) after recrystallisation from acetic anhydride and then from 95% alcohol until of constant m.p. A mixture of equal parts of this *acetyl* derivative and *cholesteryl acetate* melts at 121—122° (corr.). Sunflower seed oil also contains a small amount of a *phytosterol*, the *acetyl* derivative of which has m.p. 115.5° (corr.). The corrected m.p. of phytosteryl acetates prepared from other oils and fats are, from hydrogenated sunflower seed oil 119.25°, olive oil 123.5°, cotton seed oil 125.5°, palm kernel oil 127°, arachis oil 129°, hydrogenated arachis oil before deodorisation 128.5°, hydrogenated arachis oil after deodorisation 128°, coconut oil 129.5°, sesamé oil 129.5°, palm oil 131°, linseed oil 131°. W. J. POWELL.

Sulphonated oils and leather. BUMCKE.—See XV.

Animal skin fat. THEIS.—See XV.

PATENT.

Emulsifying agents (B.P. 258,551 and 268,387).—See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Red lead problems. I. Settling and hardening of red lead. II. Colour of red lead. III. Red lead as a composite pigment. C. P. VAN HOEK (Farben.-Ztg., 1928, 33, 981—983, 1046—1049).—A review of the literature on the causes of settling and hardening of the sediment in red lead-oil paints, and their successful overcoming in modern "dispersed" red leads. Among the many influences discussed are:—grinding, presence or absence of turpentine or other diluent, formation of lead soaps and lead glyceroxide (an important factor in hardening), dependence of this factor on free monoxide content of the pigment and free fatty acid content of the oil, particle size, and rate of settling (a slow-settling red lead usually not hardening). A somewhat full reference to Junk's work indicates that the settling and hardening cannot be attributed to one particular cause, many varying factors being jointly responsible. Microscopical examination reveals "active" and "inactive" particles, absence of the latter in "dispersed" red lead giving it its favourable non-settling properties. This activity or non-activity is accounted for by variations in the structure of the labile combination of lead monoxide and peroxide constituting the pigment. Suggestions that water is adsorbed on the surface of "dispersed" red lead are disproved. The variation in colour of red lead is discussed with reference to the colour of different varieties of lead monoxide and peroxide, the probable constitution of red lead being a lead monoxide substratum on which are adsorbed ultramicroscopic particles of lead peroxide. Microscopical examination shows that conversion of lead monoxide into red lead is not accompanied by any appreciable increase in particle size, the heterogeneous nature of the red lead particles being demonstrated under ordinary and polarised light. Various views on the composite nature of the pigment are discussed. S. S. WOOLF.

Pigment and vehicle. E. KLUMPP (Farben.-Ztg., 1928, 33, 1044—1045).—The absorption by white lead, zinc oxide, blanc fixe, and china clay of various aqueous and oily vehicles is tabulated, the value being expressed in terms of volume. The results are discussed with reference to the relation between adsorption and absorption. A white lead-linseed oil paste (67% of oil on pigment by vol.) consists of primary white lead particles separated by a thin layer of oil, but a zinc oxide-paraffin oil paste (440% of oil on pigment by vol.) consists of agglomerates or secondary particles composed of wetted primary particles. Other factors possibly tending to raise the vehicle absorption of a pigment are the presence of adsorbed gases or vapours and the formation of "vehicle-sheaths" of relatively considerable diameter around the particles. S. S. WOOLF.

Iron-cyanogen colours, in particular Milori blue. A. MÜLLER-MAGDEBURG (Chem.-Ztg., 1927, 51, 923—924).—Milori blue or steel blue is closely related to Prussian blue, and is valued for its reddish shade. It is prepared by oxidising the white paste formed by mixing sodium or potassium ferrocyanide and ferrous sulphate, the oxidising agents used being chlorine, ferric chloride, potassium dichromate and sulphuric acid, or, most commonly, potassium chlorate and hydrochloric or

nitric acid. On the large scale, using either nitric acid alone or potassium chlorate and hydrochloric acid, about 35 kg. of Milori blue are obtained from 50 kg. of potassium ferrocyanide and 45 kg. of ferrous sulphate. The chemistry of the formation and composition of Prussian blue and its related pigments is discussed. W. T. K. BRAUNHOLTZ.

W. T. K. BRAUNHOLTZ.

PATENTS.

Paint-removing composition. H. L. LEASK (B.P. 282,549, 8.12.26).—Wax, e.g., beeswax, paraffin wax, earth wax, is melted and coal-tar light oils are stirred in. Benzol and methylated spirit are finally added.

S. S. WOOLF.

Manufacture of red lead. J. A. SCHAEFFER, J. H. CALBECK, and J. R. CRENSHAW, Assrs. to EAGLE-PICHER LEAD Co. (U.S.P. 1,652,217, 13.12.27. Appl., 9.5.25. Renewed 21.10.27).—Ground litharge obtained by the oxidation of molten lead is mixed with litharge condensed from its vapour, and the mixture is oxidised in a reverberatory furnace.

T. S. WHEELER.

Manufacture of a non-dusting carbon [black] pigment. S. E. SHEPPARD and L. W. EBERLIN, Assrs. to EASTMAN KODAK Co. (U.S.P. 1,651,733, 6.12.27. Appl., 26.11.26).—Powdered carbon black is impregnated with a benzene solution of a wax, and the solvent is evaporated.

T. S. WHEELER.

Preparation of lacquers from tung oil. W. SCHMIDTING (B.P. 282,574, 28.2.27. Addn. to B.P. 247,599; B., 1926, 988).—Lacquers are prepared in an installation of three vessels located one above the other and connected by pipes, tung oil being heated in the middle vessel and additional material being run in, at a suitable stage in the cooking of the tung oil, from the upper vessel (also provided with heating apparatus). The mixture is eventually run off into the lowest vessel, where cooling and thinning take place. The outlet closure of the middle vessel is located within the vessel to avoid trapping of gelatinised oil. The three vessels are connected to a common vapour exhaust.

S. S. WOOLF.

Nitrocellulose lacquer composition. C. BOGIN, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,651,578, 6.12.27. Appl., 1.6.25).—*cyclo*Hexanol is claimed as an ingredient.

T. S. WHEELER.

Production of a composition for use in the manufacture of, or for use as, varnishes etc. H. W. MATHESON, Assr. to CANADIAN ELECTRO PRODUCTS Co., LTD. (U.S.P. 1,650,899, 29.11.27. Appl., 19.8.22).—The products obtained by heating acetylene-phenol resins with hexamethylenetetramine or paracetaldehyde are dissolved in suitable solvents.

T. S. WHEELER.

Production of condensation products of phenols with aldehydes. G. PETROFF (B.P. 283,002, 1.2.27).—Resols are obtained by condensing a phenol with an aldehyde in presence of the sodium salt of *p*-toluene-sulphonchloroamide ("Chloramine T") as catalyst instead of the usual basic or acid catalysts. The resols may be hardened by heat, or by means of acid catalysts dissolved in an alcohol, chlorinated hydrocarbon, or fatty acid, or by alkaline catalysts, dissolved as glycerol or phenol salts in an alcohol. C. HOLLINS.

Preparation of fluid condensation products from phenols and formaldehyde. G. PETROFF (G.P. 442,211, 23.7.21).—Phenols are condensed with formaldehyde in the presence of lead acetate (in amount at least 10% of the amount of phenol used), the mixture being boiled. The condensation proceeds under acid conditions, partial decomposition of the lead acetate occurring and giving rise to lead salts of "novolacs." After removal of the water layer the condensation is continued until the product has the required density. It is soluble in alcohol, benzol, and turpentine, etc., and may be blended with drying oils, natural resins, etc. S. S. WOOLF.

Manufacture of condensation products of carbamide and its derivatives and formaldehyde. I. G. FARBENIND. A.-G. (B.P. 259,950, 6.10.26. Ger., 16.10.25).—Condensation products of carbamide and its derivatives and formaldehyde are hardened by exposing them while in the gelatinous state to the action of sulphur dioxide and, if desired, heat. S. S. WOOLF.

Synthetic resin composition. H. L. BENDER, Assr. to BAKELITE CORP. (U.S.P. 1,650,109, 22.11.27. Appl., 10.11.26).—The resin formed by the action of *p*-toluenesulphonamide on formaldehyde is of value as a plasticising agent for phenol-formaldehyde resins. T. S. WHEELER.

Preparation of solvents etc. from resins. H. G. HJERPSTED (F.P. 618,740, 10.7.26. Denm., 11.7.25).—Resins are subjected to dry distillation with calcium or barium, and the distilled products are hydrogenised, e.g., in an autoclave, in the presence of catalysts, e.g., nickel, platinum, if necessary. S. S. WOOLF.

Production of resinous condensation products of the urea-formaldehyde type. ROHM & HAAS Co., Assees. of F. LAUTER (B.P. 256,248, 28.7.26. U.S., 28.7.25).—See U.S.P. 1,633,337; B., 1927, 788.

Cellulose nitrate solutions (B.P. 282,172).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Brittleness tests for rubber and gutta-percha compounds. G. T. KOHMAN and R. L. PEEK, JUN. (Ind. Eng. Chem., 1928, 20, 81–83).—India-rubber and gutta-percha compounds, e.g., insulating materials, become brittle at a definite temperature, which is characteristic of the individual material. An apparatus is described for determining the highest temperature (the "brittle temperature") at which a sample undergoes fracture when suddenly bent through an angle of 45°. The test may also be applied when the sample is under high hydrostatic pressure. The "brittle temperature" probably represents a temperature range in which the physical properties of the material undergo an abrupt change. D. F. TWISS.

Reduction of period of vulcanisation of thin dipped goods in sulphur chloride vapour. R. DITMAR and G. BALOG (Gummi-Ztg., 1928, 42, 858–859).—Vulcanisation of films (thickness 0.03 mm.) in sulphur chloride vapour (1.5 c.c. in space of 31.02 c.c.) proceeds rhythmically with alternating aggregation and dispersion in a series of successive changes. Contrary to previous views, a period of 2 min. is sufficient to give a

well-vulcanised product with good ageing. The presence of zinc oxide, lithopone, or carbon black is favourable to good ageing behaviour, but certain organic colours have an unfavourable influence. D. F. TWISS.

PATENTS.

Manufacture of compositions of matter containing caoutchouc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 282,617, 10.6.26; cf. B.P. 282,172; B., 1928, 155).—Solutions of rubber, with or without colouring matters and cellulose nitrate, are prepared using as solvent an oily product of the catalytic hydrogenation of oxides of carbon or a liquid produced by the hydrogenation, acylation, or condensation of such oily product. D. F. TWISS.

Compounding and vulcanising rubber and products obtained therefrom. O. H. SMITH, Assr. to NAUGATUCK CHEMICAL Co. (U.S.P. 1,651,737, 6.12.27. Appl. 6.7.25. Renewed 3.5.27).—Sheet rubber is dipped in benzene containing sulphur, an amine, e.g., dibenzylamine, and a metallic salt of a carbon disulphide derivative, e.g., zinc butylxanthate, and is then heated at 100° for 2 hrs. T. S. WHEELER.

Vulcanisation of rubber. C. W. BEDFORD, Assr. to B. F. GOODRICH Co. (U.S.P. 1,650,975, 29.11.27. Appl., 28.6.24).—The additive products of metallic salts, e.g., zinc sulphate, and primary amines are used. T. S. WHEELER.

Production of vulcanised rubber. H. W. ELLEY, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,652,101, 6.12.27. Appl., 2.12.26).—Cyclic thiouram mono- and di-sulphides are claimed as accelerators. T. S. WHEELER.

Renewal of india-rubber and chiefly that contained in the covers of worn-out pneumatic tyres. C. DANIER, Assr. to SYNDICAT FRANCO-NEERLANDAIS (U.S.P. 1,656,694, 17.1.28. Appl., 16.9.26. Fr., 8.4.26).—See B.P. 269,127; B., 1927, 635.

Treatment of fabrics (U.S.P. 1,651,751).—See V.

XV.—LEATHER; GLUE.

Batch B. 14 hide powder. II. D. BURTON (J. Soc. Leather Trades' Chem., 1927, 11, 386–393; cf. B., 1927, 757).—B. 14 hide powder treated with a buffer solution of *p*H 5.4 gave non-tan results closely agreeing with those given by B. 13 for liquid-blended extracts and for most liquid quebracho extracts. The non-tan values given by B. 13 were obtained with some extracts using B. 14 chromed with chrome alum. By using B. 14 chromed with a more acid chromium chloride solution, e.g., B. 14 chromed with a liquor containing 15 g. of anhydrous sodium carbonate per 100 g. of chromium chloride, results agreed most closely with those given by B. 13. Paessler's powder chromed with chrome alum gave lower non-tan values than B. 14 chromed with chrome alum or a more acid chromium chloride solution. D. WOODROFFE.

Influence of temperature on the tannin yield of pyrogallol tans. J. G. PARKER and J. A. GILMAN (J. Soc. Leather Trades' Chem., 1927, 11, 374–385).—Samples of myrobalans, valonia, sumac, and divi divi were scalded with boiling water prior to extraction, or heated for a time in an air oven at 103°, extracted, and

analysed. Both processes of preheating caused an increase in the yield of tan from myrobalans, and a diminution in the non-tans. The solutions were practically clear, showing little sign of deposition as usually occurs in the official method. Scalding overnight improved the colour of the liquors, whereas heating prior to extraction darkened the colour. The effect of both processes on *divi divi* was to decrease the tannin content and increase the insoluble matter. Scalding improved the colour and yield of tan from valonia and diminished the non-tans and insoluble matter, but was not suitable for sumac as it destroyed the tannin and increased both the insoluble matter and non-tans. Preheating caused a distinct increase in both the total soluble matter and the tans, with a slight increase of non-tans; the colour was not affected. In the official method of analysis, some hydrolysis must occur during the soaking period, which is retarded by preheating. Greater concordance in analytical results is obtained by preheating.

D. WOODROFFE.

Wattle barks. II. F. A. COOMBS, W. MCGLYNN, and M. B. WELCH (J. Proc. Roy. Soc. New South Wales, 1926, 60, 360—371).—The tannin content of wattle bark stored for thirty years was much the same as that of the original fresh bark, but the ratio of tannins to non-tannins had apparently increased, suggesting a possible increase in tannin at the expense of the non-tannins. Although the barks had become dark red in colour, there had been no apparent decrease in the solubility of the tannins. No evidence could be obtained of the existence in the old bark of fissures rendering it more readily penetrated by water. It appears probable that tannin reds do not actually occur in fresh wattle bark, but are formed in the extraction with water above 40°, which is necessary for the complete removal of the tannins, since boiling solutions of wattle tannins did not result in any loss of tannin. The losses which occur when solutions of the tannins in contact with partly spent bark are subjected to high temperatures seem to be due to the formation of a starch-tannin compound, partly soluble near the b.p., but separating out on cooling.

R. CUTHILL.

Sulphonated oils and their reaction on leather. G. BUMCKE (J. Amer. Leather Chem. Assoc., 1927, 22, 621—635; cf. B., 1915, 1214).—Two highly sulphonated cod oils *A* and *B* which proved unsatisfactory for fat-liquoring leather and two oils *C* and *D* which were satisfactory, have been analysed. The solubility in water, dark colour, and strong odour of *A* and *B* indicated that they were not made from pure Newfoundland cod oil. *C* contained a large amount of liquid unsaponifiable matter, and this reduced the other values. When the analytical data are calculated on the basis of oil free from moisture and unsaponifiable matter, the values for *C* agree with those for *A* and *B*, and indicate that the base in *C* is a fairly highly sulphonated oil, probably cod oil, though less sulphonated than *A* or *B*. The high content (5.5%) of unsaponifiable matter in *D*, excessive for a pure cod oil, might be due to shark liver, sperm, or other fish oil. It also contained a high percentage of ammonia soap (1.81%) not present in the other oils. It is suggested that the high percentages of oxidised fatty acids in *A* and *B* were responsible for

their unsatisfactory fat-liquoring properties. The base of *C* might have caused the same trouble but for the dilution with mineral oil which reduced the amount of oxidised and sulphonated fatty acids almost to the same quantity as in *D*. It is concluded that high degree of sulphonation alone or a high value for combined SO_3 is no criterion for the good quality of a sulphonated oil; further, evaluation of the oil from its so-called "sulphonation value" is quite erroneous. D. WOODROFFE.

Procter-Searle method of determining free mineral acid in leather. D. WOODROFFE (J. Soc. Leather Trades' Chem., 1927, 11, 493—397).—Procter-Searle determinations were made on samples of vegetable-tanned leathers which had been respectively treated with various products containing sulphonic groups, e.g., sulphonated oil, sulphite-cellulose, and synthetic tan. Positive results were given in every case (except that of a sulphonated vegetable oil which had been neutralised with soda) in which the sulphonated oils had been neutralised with ammonia. Samples of sulphonated cod oils, neutralised to Methyl Red, still gave positive results by the Procter-Searle method. A positive Procter-Searle value is therefore not always indicative of the presence of free mineral acid, but may be due to combined sulphuric acid. The pH value of the water extract of a leather should be determined as well as its Procter-Searle value, to ascertain whether the product in the leather is deleterious or not.

D. WOODROFFE.

The "by difference" method of determining acid absorption by pelt in one-bath chrome-tanning experiments. F. HUDSON (J. Soc. Leather Trades' Chem., 1927, 11, 398—404).—Tanning tests were made with a pure chromium sulphate, violet and green chrome alum, glucose chrome liquor, and a chromium chloride liquor. Analysis of the leather showed that the "by difference" method of determining acid absorption by the pelt from the chrome-tanning liquors was not accurate. The "total sulphate" method was also useless, since it included any sodium sulphate absorbed. This value may, however, be useful for certain control purposes.

D. WOODROFFE.

Effect of temperature on the hydrolysis of skin and hair in saturated lime water. H. B. MERRILL and J. W. FLEMING (Ind. Eng. Chem., 1928, 20, 21—23).—Samples of skin and degreased hair from calfskins were treated with saturated lime water. After 3 days at temperatures below 35° the hair was more hydrolysed than skin; above this temperature the reverse was the case. The hydrolysis of hair was a nearly linear function of lime at any temperature. The hydrolysis of skin proceeded linearly until a certain amount of decomposition had taken place, after which the digestion was very rapid. The skin was dissolved completely in a few hours at 45°, in 1—2 days at 40°, in 4—5 days at 35°, in 35—40 days at 30°, but not in 60 days at lower temperatures. These results confirm the difference in the hydrolysis of hair and skin. Hair is hydrolysed by the breaking of a primary valency linking, probably the —S—S— linking of cystine. The hydrolysis of skin is a stepwise process. Skin consists of comparatively simple polypeptide-like aggregates linked together by

secondary valency forces. During the hydrolysis of skin, the secondary valency linkings are disrupted first, and then the component aggregates are hydrolysed into progressively simpler molecules until a compound is obtained which is more soluble in hot lime water than in cold.

D. WOODROFFE.

Further characteristics of animal skin fat. E. R. THEIS (J. Amer. Leather Chem. Assoc., 1928, 23, 4—9; cf. McLaughlin and Theis, B., 1925, 602).—Portions of heavy-cured steer hide cut up into 0.125-in. cubes, were soaked for 24 hrs. and 96 hrs. at ordinary temperatures, 48 hrs. at 37.5°, 24 hrs. and limed for 120 hrs., and 96 hrs. and limed for 120 hrs., respectively. The fat was extracted in each case after the experiment and analysed. The fat from soaked hide was dark brown, and that from limed hide a light golden-yellow. The saponification and iodine values of the fat diminished with increased time of soaking, but increased sharply with liming. The amount of unsaponifiable matter was inversely proportional to the saponification value of the extracted fat. The acid value increased with the length of soak period. Steer skin fat consisted of 2% of phospholipin, 24% of solid neutral fat, and 74% of liquid neutral fat, and the fatty acids of these ingredients were not highly unsaturated. It is suggested that soaking removes the lower glycerides from animal skin fat, leaving only the higher members. Liming may possibly saponify some of the cholesteryl esters in skin, thus increasing the saponification value of the fat extracted. The decrease in iodine value during soaking is attributed to bacterial oxidation of the unsaturated fatty acids, and the increase after liming must be due to selective saponification by the lime.

D. WOODROFFE.

Behaviour of agar towards iodine. ROSENTHALER.—See XX.

PATENTS.

Preservation of hides and skins. SALT UNION, LTD., W. CLAYTON, and W. E. GIBBS (B.P. 282,128, 13.8. and 31.12.26).—0.08% of lead as lead chloride or 0.0053% of cadmium as cadmium chloride is added to a neutral natural brine (d 1.20), which may or may not be dyed, and the solution allowed to crystallise so as to produce a solid salt containing less than 1% of a lead or cadmium compound distributed throughout the crystal structure. The product is bactericidal, and is used for preserving hides and skin, thus preventing salt stains.

D. WOODROFFE.

Printing of chrome leather. ANC. ÉTABL. A. COMBE & FILS & CIE. SOC. ANON. (B.P. 256,195, 28.6.26, Fr., 31.7.25).—The colouring of chrome leather by dyeing or printing is satisfactory provided that the leather is not dried between tanning and dyeing. Chrome leather loses the greater part of its affinity for dyes when dried, and this affinity cannot be restored by steeping in water or steaming. After tanning, the chrome leather is wrung so that it contains 50—70% of moisture at the time of dyeing or printing. Alternatively, the wet leather is impregnated with an aqueous solution of glycerin and then dried so that it contains not less than 20% of glycerin; such leather behaves towards dyes similarly to leather which has been maintained wet.

A. J. HALL.

Manufacture of solutions of gelatin in organic solvents. I. G. FARBENIND. A.-G. (B.P. 279,443, 15.10.27. Ger., 20.10.26).—Gelatin is dissolved in water by the aid of an alkali or alkaline-earth hydroxide, in the absence of any other electrolyte, and an organic diluent is added; the resulting solution is alkaline. A gelatin solution of any desired p_H value may be prepared by mixing this product with a gelatin solution in which an acid has been used as peptising agent, or by addition of an organic acid.

B. FULLMAN.

Impregnation of [moulded] leather with wax etc. W. C. MATTHEWS (B.P. 282,256, 29.3.27).

XVI.—AGRICULTURE.

Study of base exchange in soils with the aid of the quinhydrone electrode. F. O. ANDEREGG and R. P. LUTZ (Soil Sci., 1927, 24, 403—412).—Using Donnan's membrane equilibrium theory, equations expressing base exchange in soils have been worked out. These have been verified by substituting the values obtained by electrometric titration of a suspension of clay "acid" (clay practically freed from cations other than hydrogen by electrodialysis) with solutions of salts in the presence of a quinhydrone electrode. The clay behaved like a monobasic acid in contact with solutions of neutral salts. The ionisation constants for the sodium, potassium, magnesium, and calcium salts of the clay are calculated, respectively as 1.70, 1.15, 0.73, and 0.76, all multiplied by 10^5 ; the exchange constants between these cations and hydrogen are also evaluated.

C. T. GIMMINGHAM.

Quinhydrone electrode and soil reaction. N. A. CLARK and E. R. COLLINS (Soil Sci., 1927, 24, 453—463).—Investigations are reported on some points in the application of the quinhydrone electrode to the measurement of p_H in soils. Various methods for preparation of the mixture of soil and water were tested, and reproducible values were obtained by stirring equal parts of soil and water, shaking for a few seconds, and allowing to settle for $\frac{1}{2}$ min., the electrode being placed in the soil paste (cf. Bühlmann and Tovborg-Jensen, B., 1927, 887). There does not appear to be a definite soil-water equilibrium, even at a ratio of 1 : 1. Differences between the p_H of the supernatant liquid and the settled soil are accentuated if the soil is passed through a colloid mill. Temperature corrections are given for the quinhydrone electrode used with the saturated potassium chloride-calomel half-cell.

C. T. GIMMINGHAM.

Comparison of the quinhydrone and hydrogen electrodes for determining the hydrogen-ion concentration of soils. E. F. SNYDER (J. Agric. Res., 1927, 35, 825—834).—Measurements of p_H values of soils varying from p_H 4.0 to 9.0 by means of a modified Gillespie electrode showed the quinhydrone electrode to be generally satisfactory. Agreement with results obtained by the hydrogen electrode was close except on the alkaline side, where irregularities were more common. Using buffer solutions, results from quinhydrone and hydrogen electrodes were in close agreement except for the range p_H 8.0—9.0. When using suspensions having a soil-water ratio of 1 : 2, equilibrium with hydrogen electrodes was usually attained in 10 min.

Substitution of palladium or iridium black for platinum in the making of the electrode was without effect when soil suspensions were examined by means of the hydrogen electrode. Gold-foil electrodes were superior to platinum in quinhydrone electrodes used with buffer solutions.

A. G. POLLARD.

Relative proportions of exchangeable bases in some Scottish soils. A. M. SMITH (J. Agric. Sci., 1928, 18, 68—75).—The changes effected by treatment of a number of soils from East Scotland with dilute solutions of sodium, potassium, and calcium chloride have been studied. The effect of 0.02*N*-solutions was very marked, the amount of exchangeable potassium in one soil being increased about 17 times by contact with 0.02*N*-potassium chloride solution. Changes caused by 0.002*N*-solutions were, however, very small, and comparable with what might be expected in practice when fertilisers are used. The content of exchangeable bases and their relative proportions vary considerably in different soils, and should prove useful as an additional characteristic of soil types.

C. T. GIMINGHAM.

Reaction, exchangeable calcium, and lime requirement of certain Scottish soils. W. G. OGG and W. T. DOW (J. Agric. Sci., 1928, 18, 131—158).—Some of the results of a survey of the soils of a wide area in South-East Scotland, including a detailed study of a single farm, are discussed. The majority of the cultivated soils had a p_H value between 5.0 and 6.5, a lime requirement of 0.05—0.25% $CaCO_3$, and an exchangeable calcium content of 0.1—0.45% CaO . There was general agreement between the three sets of figures, though with many exceptions. Woodland, hill, and heath soils were characterised by a p_H usually below 5.0, exchangeable calcium usually below 0.1% CaO , and a high lime requirement. Cultivation had frequently brought about great changes in reaction. The results support the view that Scottish soils in general belong to the podsol group. Relationships between the data given and the natural vegetation are noted.

C. T. GIMINGHAM.

Fate of fractional incorporations of burnt lime in two soil zones. W. H. MACINTIRE (Soil Sci., 1927, 24, 475—485).—Analyses are reported of the leachings from lysimeters filled with soil with which calcium hydroxide was incorporated at various rates, either in the upper or lower half (zone) of the soil. Over a period of 4 years, the losses of calcium from soil which received small additions of calcium hydroxide were practically the same as from the untreated soil. With larger amounts, losses of calcium were specially great when the lime was added to the lower zone of soil. Losses of calcium and increases of nitrates in the leachings were greatest during the first year. The relation of rate of liming to calcium conservation is discussed.

C. T. GIMINGHAM.

Distribution of phosphoric acid and potassium in soils. S. GERICKE (Z. angew. Chem., 1928, 41, 52—56).—The absorption of phosphoric acid and potassium by plants from the soil depends not only on the presence of these materials in a readily assimilable form, but also on the physical properties of the soil itself. The variation in size of the soil particles is

an important factor; this may be determined by shaking the soil with water, allowing to settle for a definite time, and pouring off the liquid containing the smaller particles in suspension, thus dividing the sample into fractions of decreasing particle size. The results obtained depend largely on the method of preparation of the analysis sample, which must be shaken or boiled with water or rubbed with a pestle to separate the particles. Phosphoric acid is found in all fractions, but chiefly in the fraction containing particles of 0.02—0.2 mm., whilst potassium salts are concentrated in the smaller particles (less than 0.002—0.02 mm.). Both substances are more readily assimilated by germinating roots from the fractions of larger particle size than from the closely packed soils of small particle size.

W. J. POWELL.

Retention of phosphorus by soil colloids. C. A. ROSZMANN (Soil Sci., 1927, 24, 465—474).—A pure colloidal clay, freed from exchangeable bases by electro-dialysis, absorbed a maximum amount of phosphoric acid, in presence of calcium or sodium salts, at p_H 3—4. There was no appreciable absorption above p_H 9. Iron and aluminium phosphates were not formed, since at the p_H at which maximum absorption occurred the salts of these metals are practically all soluble; nor was absorption due to formation of insoluble calcium salts, because soluble phosphorus only was taken into account. Some evidence is given that organic matter was responsible for some or all of the absorption of phosphorus.

C. T. GIMINGHAM.

Determination of the phosphate requirement of soils. F. TERLIKOWSKI, S. MICHNIEWSKI, and M. KWINICHIDZE (Rocz. Nauk Rolniczych i Lesnych, 1927, 17, 309—335; Bied. Zentr., 1928, 57, 8—10).—In a critical examination of methods for estimating soil fertility the importance of adapting the experimental process to suit local soil conditions is emphasised. Fresh soil samples are essential in determining phosphate requirements. Drying alters the proportion of soluble phosphate in soils to an extent peculiar to each soil. The amount of soil phosphate assimilable by plants bears no definite relationship either to the total, water-soluble, or citric-soluble phosphate present. Many soils, showing no phosphate requirement by the Mitscherlich method, were deficient according to the Némec standard. There is a definite inverse relationship between the humus content of soils and their water-soluble phosphate content.

A. G. POLLARD.

Relation of organic matter and nitrogen content to series and type in virgin grassland soils. J. C. RUSSEL and W. G. MCRUER (Soil Sci., 1927, 24, 421—452).—A study of virgin grassland soils in Nebraska indicates that texture is the chief factor determining the nitrogen content of any soil type. Textural variations can be expressed in terms of the hygroscopic coefficient, and the ratio of the coefficient to the percentage of nitrogen is used to investigate the distribution of nitrogen in various series and types. Some soil types are very homogeneous in content of nitrogen; others are decidedly heterogeneous. In a series of homogeneous types, the nitrogen content varies with the rainfall and with topography. The amounts of organic matter and nitrogen bear a fairly constant ratio to one another

both in extremes of rainfall and of texture. The application of the results to soil classification and mapping is discussed.
C. T. GIMINGHAM.

Milorganite—a new fertiliser material. V. H. KADISH (Ind. Eng. Chem., 1928, 20, 9—10).—Sewage waste of a uniform character, which is marketed at Milwaukee under this name, is a dry, evenly ground solid containing 4.08% of moisture, 86.13% of available, water-insoluble, organic nitrogen (by neutral permanganate method), and 2.43% of available phosphoric acid. It compares satisfactorily with other artificial fertilisers.

R. H. GRIFFITH.

Hot fermentation of stable manure by Krantz' process. II. GLATHE (Landw. Versuchs-Stat., 1927, 107, 65—129).—Examination of the drainage liquor from hot-fermenting manure heaps showed that the addition of peat mould to the heap not only reduced the drainage in quantity, but also lowered its content of phosphorus and potassium. During the making of ordinary cold-fermented manure the losses of nitrogen and dry matter were 2—3 times those from hot-fermented stacks. Rapidity of heating of manure stacks depended mainly on their water content and permeability to air. The latter was adequately regulated by loose stacking. Best results were obtained in hot-fermenting stacks by thorough trampling down and covering with earth immediately the maximum temperature was reached. In field trials the nitrogen of hot-fermented manure performed the same function as that from ordinary cold-fermented stacks. Its activity differed with soil type. Only in rich soils was the water-insoluble nitrogen effective on the first season's crop. Crop increases resulting from the use of hot-fermented manure were in some cases nearly twice those from cold-fermented manure. The better conservation of nutrients obtaining in hot-fermented stacks was formerly possible only by storage in deep stalls under cattle which consolidated the heap by trampling.

A. G. POLLARD.

More effective dust fungicides by the use of oxidising agents with sulphur. II. A. LEE and J. P. MARTIN (Ind. Eng. Chem., 1928, 20, 23—28).—The fungus *Helminthosporium sacchari*, which causes "eye-spot" in sugar cane, is not checked at all by such dust fumigants as Bordeaux mixture, calcium hypochlorite, and hydrated lime, and only slightly by sulphur. The addition of 1% of creolin to the sulphur, extra fine grinding, and more frequent applications caused no improvement. It was found, however, that the addition of oxidising agents to the sulphur, which promote the formation of pentathionic acid, greatly increased its efficiency. 1% of potassium permanganate as an addition reduced the disease by 90%; $\frac{1}{4}$ % of nitric acid and 5% of β -naphthol produced less improvement. These dusts also stimulated growth. Trials at another plantation on different soil gave much less successful results with these dusts. In this case Chloramine-T in a lime carrier was the most effective dust tried. Quantitative figures of degree of infection were carried out by counts of the numbers of spots per leaf.

C. IRWIN.

Differential response of barley varieties to manuring. F. G. GREGORY and F. CROWTHER (Nature, 1928, 121, 136).

PATENTS.

Manufacture of fertilisers. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (B.P. 273,291, 20.6.27. Ger., 28.6.26. Addn. to B.P. 235,860; B., 1925, 731).—The amount of silica required in the process of the parent patent is reduced to the proportion of 1 mol. of SiO_2 to 2 mols. of P_2O_5 , if sodium sulphate or chloride, or a mixture of the two, is used as the alkali salt. If the raw phosphate contains calcium not combined with phosphoric acid (e.g., as calcium carbonate or fluoride), sufficient silica must be added to convert this calcium into calcium orthosilicate. C. T. GIMINGHAM.

Fertiliser. SYNTHETIC AMMONIA & NITRATES, LTD., and A. E. MITCHELL (B.P. 282,463, 19.6.26).—Powdered alkali sulphate, preferably potassium sulphate, added to a mixture of ordinary crumbled superphosphate and powdered ammonium nitrate yields a mixed fertiliser which does not rot the bags in which it is stored nor corrode metal. By adjusting the proportions of the ingredients, a fertiliser containing predetermined amounts of nitrogen, phosphorus, and potassium can be prepared; as little as 2% of added sulphate gives a satisfactory mixture.

C. T. GIMINGHAM.

Production of a fertiliser product. G. BARSKY, ASSR. to AMER. CYANAMID CO. (U.S.P. 1,651,363, 6.12.27. Appl., 29.12.23).—An intimate mixture of calcium cyanamide and phosphate rock is treated with sulphuric acid.

T. S. WHEELER.

Manufacture of briquettes containing phosphate rock. J. A. BARR, ASSR. to INTERNAT. AGRICULTURAL CORP. (U.S.P. 1,655,981, 10.1.28. Appl., 9.4.24).—About 6 pts. of ground phosphate rock are mixed with 2 pts. of powdered coal and 2 pts. of silica, and the product is made into briquettes and heated at approximately red heat. The final product is substantially waterproof.

H. ROYAL-DAWSON.

Insecticide and method of applying same. W. L. TANNER, ASSR. to GRASSELLI CHEMICAL CO. (U.S.P. 1,652,291, 13.12.27. Appl., 1.10.21).—A sublimable, solid, organic, trivalent arsenic compound, e.g., phenarsazine chloride, is packed in a container with a core of celluloid, which is ignited to vaporise it.

T. S. WHEELER.

XVII.—SUGARS; STARCHES; GUMS.

Barium compound of technical interest from starch. E. STERN (Z. angew. Chem., 1928, 41, 88—91).—The precipitation of starch by means of its barium oxide compound avoids the necessity of evaporating large quantities of water in preparing soluble starches and starch pastes. The solutions obtained by treating starch with dilute sodium hydroxide solutions yield the whole of their starch content as a precipitate on addition of the equivalent quantity of barium chloride; the precipitate is readily collected on a filter and dried, and on treatment with a soluble sulphate (e.g., sodium or aluminium sulphate) in presence of water regenerates the soluble starch compound with precipitation of barium sulphate. The probable compositions of the various starch preparations and compounds are discussed.

S. I. LEVY.

PATENTS.

Manufacture of fructose [lævulose] from inulin. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 272,876, 23.5.27. Ger., 15.6.26).—Aqueous inulin pulp is heated with volatile organic acids, especially formic (also acetic, carbonic, etc.). The resulting syrup, treated with charcoal and concentrated *in vacuo*, yields pure crystalline lævulose, the acid being simultaneously driven off.

B. FULLMAN.

Starch conversion products. W. D. STEIN (B.P. 282,178, 28.9.26).—Substantially dry starch (containing, *e.g.*, not more than 12% of water) is treated with acetic acid (or with a salt such as calcium hydrogen acetate, which evolves acetic acid on heating) and a small amount of a catalyst, *e.g.*, ammonium persulphate, and heated with agitation to about 163°. The product contains about 13% of unaltered starch; 62% of amylolysis products, including mono- to hexa-acetate of starch, and tri-, tetra-, and hexa-amylose; 20% of sugars, *e.g.*, dextrose, maltose, raffinose, etc.; and about 0.4% of acid. When present in flour (1–20%) it improves the baked products by increasing the volume and preventing "ropiness" (by increasing acidity).

B. FULLMAN.

XVIII.—FERMENTATION INDUSTRIES.

Preservative principles of hops. VIII. Modification of the gravimetric method for the evaluation of hops. J. J. H. HASTINGS and T. K. WALKER (J. Inst. Brew., 1928, 34, 9–13).—10 g. of minced hops are extracted for 3 hrs. in a Soxhlet apparatus with 200 c.c. of ether which should not be heated above 55°. After removal of the ether by distillation, the last portion being removed under diminished pressure, the resinous residue is extracted with successive portions (20 c.c.) of warm (50–60°) methyl alcohol, and the mixed extracts are made up to 100 c.c. and filtered. To determine the α -resin, the α -resin lead salt is precipitated at 60° in 40 c.c. of the filtrate by the addition of a 1% solution of lead acetate in methyl alcohol, an excess of 1–2 c.c. of the lead acetate solution being permissible. After keeping for 30 min., the lead salt is collected on a filter in a tared Gooch crucible, washed, and dried at 100° for 1 hr. Then, weight of α -resin lead salt $\times 15.75 = \%$ of α -resin. The total soft resins are determined by diluting 20 c.c. of the remainder of the methyl alcohol solution with 40 c.c. of water containing a pinch of sodium chloride. The resins are extracted with four successive volumes, each of 50 c.c., of light petroleum (b.p. below 40°), and, after distillation of the petroleum from the combined extracts, the residue is dried at 100° for 1½ hrs. The weight of total soft resins $\times 50 = \%$ of total soft resins. The content of β -resin is obtained by difference from the total soft resins and the α -resin. (Cf. B., 1927, 24.)

C. RANKEN.

Extraction of β -resin in the determination of hop resins. A. H. BURGESS and H. MARTIN (J. Inst. Brew., 1928, 34, 13–15).—Extraction of β -resin from the methyl alcohol solution by light petroleum, although uninfluenced by an excess of sulphuric acid, is affected by the amount of water present. The quantity of

resin extracted increases until the ratio of methyl alcohol to water reaches 20 : 15, after which a decrease takes place. The additional resin extracted after dilution of the methyl alcohol possesses preservative properties.

C. RANKEN.

Yeast cell and yeast cake. N. C. BEETLESTONE (J. Inst. Brew., 1928, 34, 22–38).—The moisture in the yeast cell has been determined experimentally and by calculation. Errors due to the effects of diffusion through the cell walls are negligible and necessitate no correction in the determination of the cell moisture. In the experimental methods, which are modifications of the original "dilution" method, the quantity of drainings present in the yeast mass is ascertained from the amounts of alcohol contained in the yeast mass and the drainings. This value, together with the sp. gr. and the residual gravity of the drainings, and the dry solids of the yeast, permit the calculation of an average moisture content in the yeast cell of 51.6%. Using results published by Tait and Fletcher, the average moisture of the cell is calculated to be 47%, and by adapting the formula devised by Hehner and Richmond for the determination of the total solids of milk, a value of 58.7% is obtained. From the various figures, the moisture of a normal yeast cell is finally chosen as 54%. Yeast cake, however dry it may appear, contains free liquid varying according to the method of separation of the yeast. Under ideal conditions the pressed cake, which consists of undistorted cells arranged in hexagonal packing, contains approximately 74% by vol. of cells and retains in the intracellular space approximately 26% by vol. of the liquid from which the cells have been separated. The cells may be distended as a result of hydrostatic or osmotic pressure.

C. RANKEN.

Water content of yeast cells in suspension in brewery worts. R. H. HOPKINS (J. Inst. Brew., 1928, 34, 39–41).—Assuming that 0.35% is the average quantity of yeast increase (as dry solids) during fermentation, that 60% of those solids consists of nitrogenous and mineral matter, that 1 g. of yeast solids utilises 1.19 g. of maltose by assimilation, and that the differences in degrees of gravity lost, for a definite spirit indication, are proportional to the yeast increase, expressions are deduced for the original sp. gr. of the yeast cells and for the difference between the degrees of gravity lost, for 6° of spirit indication, with worts of 1045° and 1099° original gravity. By this method, it is calculated that the original gravity of the yeast cell is 1270°, and that 0.35 g. of yeast solids is associated with 0.52 g. of water, from which it follows that 60% of water is present in the yeast cell suspended in wort.

C. RANKEN.

Alcohol content of wash-water of carbonic acid from closed fermentation vessels. E. LÜHDER (Z. Spiritusind., 1928, 51, 1–2).—Since the greater part is condensed in the upper part of the fermentation vessel, the amount of alcohol carried over is small. In most cases it is only about 0.4% of the total alcohol produced. The variation with volume of liquid in the fermentation vessel is illustrated by a test in a 10,000 litre vessel, using 3000, 6000, and 9000 litres, when 400 litres of wash-water afterwards contained 0.0, 0.8, and 1.5% of alcohol, respectively.

F. E. DAY.

Oxalic acid and its adsorption compounds in beers. C. GEYS (Woch. Brau., 1928, 45, 1—7).—The association of calcium oxalate with the flocculent sediment of beers is very intimate, probably owing to adsorption of oxalate ions by the colloids. The greater part of the oxalic acid is derived from the malt, and as acidity increases during fermentation the colloids tend to coagulate, calcium oxalate crystals being formed in association with the flocks. Conditions such as warmth, shaking, or presence of gas bubbles, which favour flocculation, in general increase crystal formation, which is inhibited by heating the beer, as the incipient crystals which act as centres of crystallisation are dissolved. Pale beers deposit more oxalate than dark beers, since their colloids are in a higher state of dispersion and hydration and more readily adsorb the oxalate ions. The approximate solubility of calcium oxalate in distilled water, pale wort (12%), pale new beer (12%), and pale finished beer (12%) is given as 6.6, 60, 30, and 20 mg. per litre, respectively. F. E. DAY.

Identification of lactic acid. GRÜSS.—See III.
Fermentation of stable manure. GLATHE.—See XVI.
Water for brewing. STRELL.—See XXIII.

PATENTS.

Increasing the activity of yeast and activator for yeast. A. W. HIXSON and A. K. BALLS, Assrs. to FLEISCHMANN Co. (U.S.P. 1,651,027, 29.11.27. Appl., 12.12.22).—The yeast prior to incorporation in the dough is stirred for 15 hrs. at 20° with an aqueous solution of sodium stearate containing 6.7×10^{-7} — 4×10^{-5} g.-mol./g. of yeast. T. S. WHEELER.

Production of acetone and butyl alcohol by fermentation. E. F. PIKE and H. F. SMYTH, Assrs. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,655,435, 10.1.28. Appl., 29.5.23).—A culture of *Clostridium butyricum* is added in the absence of amylaceous materials to a sterilised mash of molasses and water, and the fermentation allowed to proceed to completion. C. RANKEN.

XIX.—FOODS.

Importance of particle size in manufacture of flour and baking. G. VAN DER LEE (Chem. Weekblad, 1928, 25, 34—40).—Very fine grinding of flour is not practised by reason of the cost, the danger of heating, and the difficulty of sifting. Silk gauze does not give accurate separation according to particle size, the material passing the sieve being much finer than corresponds to the size of the holes. The finer fractions have lower nitrogen content and higher diastatic power; the medium fractions are more suitable for baking than the fine and the coarse, but this is due to the differences in chemical composition rather than to particle size. The differences in suitability for baking are, in any case, small; where an unsuitable flour is found to be finely ground, the overheating in grinding, rather than the fineness attained, is found to be the determining cause. S. I. LEVY.

Method of humanising cow's or goat's milk. O. FERRIER and V. CHENARD (J. Pharm. Chim., 1927, [viii], 6, 562—563).—To 500 c.c. of cow's or goat's milk diluted with 500 c.c. of boiled water there are added

45 g. of lactose and variable small quantities of tricalcium and magnesium phosphates, disodium hydrogen phosphate, iron glycerophosphate, and sodium fluoride. Cream or fresh butter (25 g.) is added, and sometimes cod-liver oil etc. The mixture is boiled for 15 min. It is efficacious in the case of infants suffering from gastro-enteritis. B. FULLMAN.

Bromocresol Green as indicator in the manufacture of grain-curd casein. A. G. BENTON (Ind. Eng. Chem., 1928, 20, 15).—Control of hydrogen-ion concentration is essential in the preparation of technical casein, and Methyl Red has been generally used for this purpose. As this indicator is not stable, the substitution of Bromocresol Green, of which standards can be preserved much longer, is suggested. R. H. GRIFFITH.

Destruction of essence of mustard in its preparations. E. LASAUSSE (J. Pharm. Chim., 1927, [viii], 6, 97—107).—Fresh mustard preparations contain no free sulphur, but a gradual decomposition of the allylthiocarbimide takes place on keeping with liberation of sulphur. This reaction also occurs in aqueous-alcoholic solution in the cold after a few hours, and more rapidly on heating. When determining the essence content of a mustard preparation in the usual way by slowly distilling the aqueous-alcoholic solution into ammoniacal silver nitrate solution, part of the free sulphur present is carried over with the distillate and forms silver sulphide, thus increasing the apparent content of allylthiocarbimide. No liberation of sulphur from the allylthiocarbimide occurs during the actual process of distillation, probably because the bulk of this essence distils over in the early stages, and consequently accurate results are obtainable by this method when applied to freshly made mustard preparations. F. R. ENNOS.

Necessity for the evaluation of mustard flour. LEBRUN (J. Pharm. Chim., 1927, [viii], 6, 109—110).—Four samples of mustard flour, stated to conform to a standard of 0.70% of allylthiocarbimide, on examination were found to contain 0.37, 0.32, 0.64, and 0.838%. F. R. ENNOS.

Preservative action in catsup of salt, sugar, benzoate, and acid. C. S. PEDERSON and R. S. BREED (New York Agric. Exp. Sta. Bull., 1926, No. 538, 3—15).—Sugar is ineffective. Sugar (15%) with salt (3.5%) is effective. Acetic acid (1.0%) with salt (5.0%) and sodium benzoate (0.2%) stops the growth of all the bacteria tested. CHEMICAL ABSTRACTS.

Relation of *d*-gossypol to the toxicity of some cotton-seed products. W. D. GALLUP (Ind. Eng. Chem., 1927, 20, 59—63; cf. Sherwood, B., 1926, 564; Gallup, 1927, 883).—During the heating of cotton-seeds the gossypol is converted into *d*-gossypol, which is insoluble in ether. The extent of the change depends on the temperature, time of heating, and the amount of water present, and has been observed chemically and pharmacologically over a period of 16 hrs. *d*-Gossypol is toxic but less so than gossypol, and the toxicity of the seeds decreases with heating. Continued heating, especially in the presence of excess of water, destroys the *d*-gossypol, finally giving a product free from both forms of gossypol and almost non-toxic. Cottonseed meal

contains only traces of gossypol but relatively large amounts of *d*-gossypol, to which the toxicity of the meal is due. Heating the meal in the presence of water destroys the toxic properties. The insoluble gossypol formed by heating the dry seeds is much more toxic than the insoluble form found in the meal or in seeds which have been steam-heated. The existing chemical methods for the determination of the gossypols are not reliable for measuring the toxicity of cotton-seed products.

E. H. SHARPLES.

Tinplate. KOHMAN and SANBORN.—See X.
Coconut oil in cocoa preparations. HÄRTEL.—See XII.

PATENTS.

Manufacture of artificial sausage skins. J. SAMUEL (U.S.P. 1,656,681, 17.1.28. Appl., 21.9.25. Ger., 7.8.25).—See B.P. 262,202; B., 1927, 171.

Manufacture of thick gravies. E. M. ERSLEV (JØRGENSEN) (B.P. 282,475, 17.5.27).

Mixing apparatus (B.P. 281,904).—See I. **Starch conversion products** (B.P. 282,178).—See XVII. **Increasing activity of yeast** (U.S.P. 1,651,027).—See XVIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Detection of arsenic in [medicinal] iron preparations by means of hypophosphite. G. WALLRABE (Pharm. Zentr., 1928, 69, 33—37).—The employment of Thiele's reagent, obtained by treatment of sodium hypophosphite with concentrated hydrochloric acid, in place of stannous chloride in the detection of arsenic in medicinal preparations of iron compounds, as recommended by the new German pharmacopœia, is found to lead to less accurate results, and the new test is described as definitely less sensitive. Different results are obtained with different iron preparations; this is traced back to the presence of varying proportions of ferrous and ferric compounds, and it is established that ferric compounds are not completely reduced, even by large excess of Thiele's reagent. The addition of stannous chloride or of potassium iodide increases the sensitiveness of the test.

S. I. LEVY.

Chemical characteristics of drugs. Behaviour of agar towards iodine. L. ROSENTHALER (Pharm. Ztg., 1928, 73, 77—78).—A series of experiments on the reaction of agar in aqueous suspension towards iodine is described. The colour produced is dependent on the amount and concentration of iodine solution, the age, temperature of heating, temperature of the agar solution after cooling, and the method of addition of the iodine solution. The following method is suggested: 0.1 g. of powdered agar and 50 c.c. of water are boiled for 5 min.; 10 c.c. of this liquid are rapidly cooled (under 3 min.) to 15°, and 1 c.c. of 0.05*N*-iodine solution is added all at once; a bluish-violet colour is formed. The remainder of the solution is cooled to 15° and, after 1 hr., 10 c.c. are mixed with 1 c.c. of the iodine solution; no coloration should be formed.

E. H. SHARPLES.

Bactericidal power of some preparations of iodine. P. SUIFFET (J. Pharm. Chim., 1927, [viii], 6, 398—401).—The bactericidal power of various iodine solutions, ointments, etc. is determined by the time taken to kill *B. pyocyaneus*, *B. coli*, staphylococci, and streptococci. The iodine content of the preparations was 0.005%, but of the oils and ointments 0.1%. *B. coli* is the most vulnerable, followed in order by streptococci, staphylococci, and *B. pyocyaneus*. Aqueous iodine has the highest bactericidal power, followed by iodine in potassium iodide. Then follow in order tincture of iodine with water, and iodinated preparations of gum, tannin, starch, agar-agar, glycerol, and gelatin. The power of preparations of olive oil, vaseline, etc. depends on their iodine content.

B. FULLMAN.

Titration of ipecacuanha extract. E. LÉGER (J. Pharm. Chim., 1927, [viii], 6, 501—505).—Ipecacuanha extract is dissolved in water, treated with powdered pumice, and the mixture evaporated to dryness and powdered. The powder is digested with alcoholic ammonia solution, ether is added, and the digestion continued. The liquid is filtered (the volume *V* of the filtrate being noted) and distilled, and the residue dissolved in acidified water, precipitated by ammonia, and extracted with chloroform. The residue left on distillation of the latter is purified by repeated treatment with ether and distillation. The residue is dissolved in alcohol, water is added, and the liquid titrated with 0.1*N*-sulphuric acid (hæmatoxylin as indicator). From the quantity of acid used and the factor *V* the percentage of alkaloids is calculated. Alternatively, the residue is dissolved in 0.1*N*-acid and titrated with potassium hydroxide (Methyl Red). Determinations should be referred to the dry extract, which should contain 8—10% of alkaloids.

B. FULLMAN.

Insolubility of adrenaline in oils. F. MARTIN (J. Pharm. Chim., 1927, [viii] 6, 248—250).—Adrenaline base is insoluble in vaseline and in olive and castor oils. A stable, homogeneous emulsion of adrenaline hydrochloride suitable for therapeutic use may be prepared by dissolving 1 g. of adrenaline in 10 g. of alcohol with the aid of the smallest possible quantity of hydrochloric acid, adding the solution to 100 g. of castor oil, and making the mixture up to 1 litre by the addition of olive oil.

E. H. SHARPLES.

ψ-Ephedrine from *Ephedra alata*. O. F. BLACK and J. W. KELLY (Amer. J. Pharm., 1927, 99, 748—751).—ψ-Ephedrine was obtained in about 1% yield by extracting the dried twigs of *E. alata*, from Morocco, with chloroform.

S. COFFEY.

Production of ergot extract. RAPP and R. LECHLER, JUN. (Pharm. Ztg., 1928, 73, 76—77).—From experiments with pure ergotamine it is shown that, in the neutralisation of the alcoholic extract previous to extraction of the alkaloids, if a *p_H* value exceeding 7.7 is attained there is a large loss of ergotamine. Neutralisation to *p_H* 8.0 caused a loss of 18% of the alkaloid. Thus litmus and phenolphthalein are not satisfactory indicators for the neutralisation, and the authors suggest the use of a Folien colorimeter. For the extraction of ergot, a

modified percolation method, which is shorter, gives a higher yield, and decreases the volume of solvent necessary, is described. E. H. SHARPLES.

Terpeneless lemon oil. W. G. DALTON (Perf. Ess. Oil Rec., 1928, 19, 7—8).—From the examination of a large number of genuine terpeneless lemon oils, the following limits for the physical and chemical constants are given; $d_{4}^{15.5}$ 0.8935—0.8980, n_D^{20} 1.4807—1.4820, α —3° to —8°, citral 42—52%, esters 18—22%; soluble in 80% alcohol in all proportions. The aldehyde/ester ratio in all the samples examined was roughly 5 : 2, and all the oils were soluble in 4—5 vols. of 78% alcohol, but in no case was any sample soluble in 75% alcohol. The addition of citral from lemongrass oil together with an appropriate amount of linalyl acetate rendered the oil soluble in 75% alcohol, even when the original oil contained a comparatively low percentage of aldehydes. The values given by four commercial oils, all of which contained lemongrass citral, did not agree with the above specification. The significance and detection of added lemongrass citral in terpeneless lemon oils is discussed.

E. H. SHARPLES.

Lignite generator tar and oil of camomile. RUHEMANN and LEWY.—See II.

Determination of sodium borate. FRANÇOIS and SEGUIN.—See VII.

PATENTS.

Manufacture of compounds [salts] of acetamidosalicylic acid with aliphatic amines. G. W. RAIZISS and A. PROSKOURIAKOFF, ASSRS. to ABBOTT LABORATORIES (U.S.P. 1,652,796, 13.12.27. Appl., 15.10.25).—The acetamidosalicylic acid, *e.g.*, 5-acetamidosalicylic acid, is dissolved in an alcoholic solution of the amine, *e.g.*, *n*-propylamine or hexamethylenetetramine, and the salt is precipitated with ether. T. S. WHEELER.

Manufacture of cyclohexylresorcinol. M. PHILLIPS (U.S.P. 1,650,036, 22.11.27. Appl., 28.4.26).—A cyclohexane derivative containing a negative substituent, *e.g.*, cyclohexanol, is heated with resorcinol at 150° in presence of a condensing agent, *e.g.*, zinc chloride, to give cyclohexylresorcinol, m.p. 124°. T. S. WHEELER.

Manufacture of physiologically-active substances from female internal secretive organs. M. HARTMANN, ASSR. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,651,770, 6.12.27. Appl., 22.1.27. Switz., 2.2.26).—See B.P. 265,567; B., 1928, 107.

Electrode for therapeutic application (U.S.P. 1,655,783).—See XI.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENTS.

Obtaining photographic images [light-sensitive diazo compounds]. E. GAY (B.P. 282,894, 30.9.26).—The diazo compounds of ethers of *o*-aminophenols or *o*-aminonaphthols are suitable for production of light-sensitive layers on paper etc. Examples are: *o*-anisidine (developed with 2 : 4-tolylene-diamine for orange-brown),

o-phenetidine (developed with phloroglucinol for deep brown), cresidine (developed with 2-amino-8-naphthol-3 : 6-disulphonic acid for purple-brown), and, in particular, 2-aminoquinol dimethyl ether [2 : 5-dimethoxy-aniline] (developed with 1-amino-8-naphthol-3 : 6-disulphonic acid for violet). C. HOLLINS.

Photographic and etching process and product. M. C. BEEBE, ASSR. to WADSWORTH WATCH CASE CO. (U.S.P. 1,655,127, 3.1.28. Appl., 28.8.25).—A dyed substratum for applying between the sensitive coating and the metal plate consists of a solution of about 35 g. of dye in about 1300 g. of alcohol containing a slow-drying constituent. W. CLARK.

Reduction of photographic silver image layers. P. PLAGWITZ, ASSR. to E. TAESCHNER CHEM.-PHARM. FABR. (U.S.P. 1,656,235, 17.1.28. Appl., 6.5.26).—See B.P. 260,892; B., 1927, 61.

[Production of offset printing plates for] photo-engraving processes. DEPTH-O-TONE CORP., ASSEES. of F. STOCKINGER (B.P. 271,468, 16.5.27. U.S., 18.5.26).

Carbon pigment (U.S.P. 1,651,733).—See XIII.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

[Permitted] explosive. H. H. CHAMPNEY, ASSR. to HERCULES POWDER CO. (U.S.P. 1,650,186, 22.11.27. Appl., 8.5.25).—A mixture of nitroglycerin, ammonium nitrate, and ground oat husks is claimed.

T. S. WHEELER.

Production of a cartridge for use with liquid oxygen. C. MOTT, ASSR. to PUROX CO. (U.S.P. 1,650,766, 29.11.27. Appl., 13.8.23).—Dehydrated molasses mixed with powdered charcoal (1—3 pts.) is employed.

T. S. WHEELER.

XXIII.—SANITATION; WATER PURIFICATION.

Testing a carbon monoxide gas-mask. F. HEINRICH and E. PETZOLD (Chem. Fabr., 1928, 19—22).—The Degea gas-mask for use in atmospheres containing carbon monoxide consists essentially of a filter impregnated with a catalyst, which promotes the oxidation of the carbon monoxide to dioxide by the oxygen in the air, and is provided with an indicator which liberates acetylene as a warning when the efficiency of the filter begins to be impaired. In air containing 1.5% CO the life of the filter is about 40 min., and in air containing 4.4% about 10 min. To test the efficiency of the mask the gases are passed through at a definite velocity, thence through potassium mercuric iodide to remove acetylene, concentrated sulphuric acid to absorb hydrocarbons, calcium chloride and phosphorus pentoxide to remove moisture, and soda-lime and phosphorus pentoxide to absorb carbon dioxide, and finally over copper oxide at 300°, any carbon dioxide formed being absorbed in soda-lime after drying in the usual way. For qualitative detection of minute traces of carbon monoxide the blood test gives the most dependable

results; the ammoniacal silver solution and palladous chloride tests are uncertain in the presence of hydrogen, acetylene, and carbon dioxide. A. R. POWELL.

Germicidal efficiency of sodium hydroxide and sodium hydroxide-carbonate mixtures at the same hydrogen-ion concentration. M. LEVINE, E. E. PETERSON, and J. H. BUCHANAN (Ind. Eng. Chem., 1928, 40, 63—65; cf. B., 1928, 70).—The germicidal efficiency of different solutions was measured by the time required to kill 99.9% of the spores of a given bacterium. At p_H 13.0—13.2 the presence of sodium carbonate increased the efficiency, but at p_H 13.2—13.4 it diminished it. The p_H value is, therefore, not a measure of germicidal efficiency, neither is the alkalinity by titration with Methyl Orange. The difficulty of the accurate determination of p_H value at high alkalinity is pointed out, and it is suggested that the determining factor in germicidal action may be undissociated sodium hydroxide. C. IRWIN.

Scientific sewage disposal at Milwaukee. R. CRAMER and J. A. WILSON (Ind. Eng. Chem., 1928, 20, 4—9).—A brief description of this activated-sludge plant (diffused air system) and its operation, including sludge disposal, is followed by a discussion of the mechanism of the process, which is claimed to be essentially non-bacterial. A comparison of the various reagents used for "conditioning" the sludge prior to filtration indicates the superiority of ferric chloride, reasons for which are discussed. The lines of present and future research are indicated. C. JEPSON.

Odours and sewage sludge digestion. I. Effect of sea water on hydrogen sulphide production. W. RUDOLFS and P. J. A. ZELLER (Ind. Eng. Chem., 1928, 20, 48—50).—Sea water is shown to retard but not prevent the digestion of sewage sludge. The presence of hydrogen sulphide in the gaseous products is due to the utilisation of sulphates present in sea water, though less than 6% of the sulphur utilised was recovered in this form. C. JEPSON.

Coagulation studies [in water purification] at the Washington suburban sanitary district. R. B. MORSE, C. A. HECHMER, and S. T. POWELL (Ind. Eng. Chem., 1928, 20, 56—59).—The water supply is treated at two rapid sand-filtration plants, one at Hyattsville and the other at Burnt Mills. At the former it was found that with a constant addition of 0.2 grain of sodium aluminate per gallon the alum, lime, and soda ash to be materially reduced with a slight saving in cost but a manifest improvement in the quality of water supplied. At Burnt Mills, which is the larger of the two plants, the p_H value of the raw water is usually higher, and apparently little advantage is to be gained by the use of sodium aluminate except at times of high turbidity. C. JEPSON.

Effect of salts on rate of coagulation and optimum precipitation of alum floc [in water purification]. B. H. PETERSON and E. BARTOW (Ind. Eng. Chem., 1928, 20, 51—55).—The salt content of the water to be treated is more of a controlling factor governing floc production than the hydrogen-ion concentration. Sul-

phates reduce the time required to produce the alum floc and extend the p_H range over which this may occur. Oxalates and phosphates probably form complex compounds which have properties different from the true floc. C. JEPSON.

Biological purification of water for brewing. M. STRELL (Woch. Brau., 1927, 44, 607—613).—The micro-organisms can be removed by filtration or destroyed by chemical or electrochemical methods. Restricted supplies of water may be purified by Berkefeld, Böhling, Delphin, Chamberland, Sukro, or Seitz filters, which differ only in the composition of the filter material. The absolute removal of the bacteria cannot be guaranteed, although the pores in such filters, which vary between 0.41 μ and 0.16 μ in diameter, are smaller than the bacteria. Sand filters are used for greater volumes of water. The removal of the bacteria is effected in the uppermost sand layer and in the filter film, which forms during filtration from animal, plant, and mineral debris. Bacteria smaller than 2 μ —0.5 μ would not be kept back but for the filter film. To ensure absolute purity, two filters in series are used, or the water is sterilised by chemical means. In the chemical method part of the water is treated with chlorine gas or ozonised air, and subsequently mixed with the remainder of the water. A treatment of 0.2—0.3 g. of chlorine per m.³ of water is sufficient. The apparatus for purification of the water by electro-osmosis is built up of a series of cells, each divided into three compartments by porous diaphragms, the electrodes being hung in the outer compartments. The water flowing through the inner compartments is electrolysed, and the ions derived from the salts in solution pass through the diaphragms into the outer compartments, with the result that not only are the bacteria destroyed by the nascent ions, but the hardness may also be wholly or partly removed from the water. From 50 to 100 litres per hour of water free from bacteria and salts can be obtained from a water giving 400 g. of residue per m.³, and having a hardness of 12.4° by means of a current of 30 kw.-hrs. C. RANKEN.

Milorganite. KADISH.—See XVI. **Bactericidal power of iodine preparations.** SUFFET.—See XX.

PATENTS.

Refuse-consuming furnaces. WOODALL-DUCKHAM (1920), LTD., E. W. SMITH, and J. W. REBER (B.P. 282,262, 13.4.27).—The refuse is burnt in a shaft furnace (with blast), which increases in diameter upwards, and the material is fed through a sleeve which can be lowered more or less into the shaft, thus regulating the upper level of the refuse. B. M. VENABLES.

Purification and softening of water. W. PATERSON (B.P. 282,165, 17.9.26).—The water is treated with an excess of caustic lime and then carbonated before passing through a zeolite bed, by adding carbon dioxide directly or as a solution of predetermined strength. The deleterious effect on zeolites of caustic lime and fine particles of calcium carbonate is thus avoided.

W. G. CAREY.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 16, 1928.

I.—GENERAL; PLANT; MACHINERY.

Chemical industries and Merseyside. A. HOLT (J.S.C.I., 1927, 46, 439—444 T).—The paper gives a brief *résumé* of the history and development of chemical industries in South West Lancashire and on the banks of the Mersey from early times till about the commencement of the present century.

Scattering of light by graded particles in suspension. T. M. LOWRY and M. C. MARSH (J. Oil and Colour Chem. Assoc., 1928, 11, 3—12).—The light-transmission of suspensions in medicinal paraffin of various sized particles of barytes (graded by elutriation) is recorded. Optical density is proportional to surface-concentration of the powder, but increases as the diameter is reduced. The probable existence of a maximum degree of covering power on continued subdivision is indicated. S. S. WOOLF.

Measurement of particle size in ground powders. J. V. RAMSDEN (J. Oil and Colour Chem. Assoc., 1928, 11, 16—17).—A new projecting microscope is described and illustrated. A very dilute aqueous suspension of the material under test is poured into a transparent dish, which is placed on the stage of an inverted microscope. The image of the particles is projected on to a calibrated screen or photographic plate. All motions of the instrument are on the Kelvin slide system. S. S. WOOLF.

Production of fibrous precipitates. VON WEIMARN.—See V. **Measurement of plasticity.** TALWALKAR and PARMELEE.—See VIII. **Recording Soxhlet extractor.** RANKOW.—See XII.

PATENTS.

Furnace. T. A. MARSH, ASSR. to GREEN ENGINEERING Co. (U.S.P. 1,656,643, 17.1.28. Appl., 29.3.23).—The grate travels between the side walls of the furnace, and pipes of uniform diameter conveying cooling fluid extend the full length of one side of the grate. The forward ends of these pipes are continued past the ignition zone of the fuel bed on the grate and are bent outwards from the grate in advance of the zone to prevent their cooling the zone. The remaining portions of the pipes extend to the rear of the grate mainly in direct contact with the fuel bed. H. HOLMES.

Kiln. H. G. LYKKEN (U.S.P. 1,656,642, 17.1.28. Appl., 15.4.25).—The kiln comprises a floor supporting the goods to be burned, side walls, and a roof. Combustion gases are supplied to the goods under pressure and escape through adjustable openings provided in the side walls above the floor. H. HOLMES.

Dryer. W. C. CARPENTER (U.S.P. 1,655,724, 10.1.28. Appl., 3.12.25).—Material to be dried is delivered into

one end of a rotary drum and discharged from the other end. Ducts for hot air extend along the interior wall of the drum, and their discharge ends are bent back to direct the air through the drum between the ducts.

H. HOLMES.

Centrifugal machine. W. C. LAUGHLIN, ASSR. to LAUGHLIN FILTER CORP. (U.S.P. 1,655,426, 10.1.28. Appl., 23.2.26).—A distributor with a helical flange is mounted within a screen carried by a rotary drum. Blades mounted loosely on the periphery of the flange are maintained in continuous contact with the screen surface solely by centrifugal action. H. HOLMES.

Adjustable plough for centrifugal grinding mills. W. A. GIBSON, ASSR. to BRADLEY PULVERISER Co. (U.S.P. 1,655,499, 10.1.28. Appl., 29.12.24).—A centrifugally-operated grinding member carrying a plough co-operates with an annular die, and means are provided for adjusting the inclination of the plough to the plane of the pan about an axis radial to its direction of movement. H. HOLMES.

Crusher. W. E. KEINE, ASSR. to ALLIS-CHALMERS MANUF. Co. (U.S.P. 1,656,004, 10.1.28. Appl., 17.5.26).—A crushing head co-operating with a concave surface is gyrated by an eccentric rotatable on a fixed vertical support and driven by a motor mounted co-axially above the support. H. HOLMES.

Mixing machine. W. F. DEHUFF (U.S.P. 1,656,665, 17.1.28. Appl., 23.3.27).—A beater shaft is supported for movement around a driving shaft parallel to it. The former carries an internal gear, engaged by a gear on the driving shaft, and also a second gear engaging a stationary gear. H. HOLMES.

Facilitating the removal of a substance solidified or condensed on cooling drums. APPAREILS ET EVAPORATEURS KESTNER (B.P. 275,952, 19.7.27. Fr., 10.8.26).—A layer of finely-divided dry powder is applied to the drum and forms a non-adherent parting layer so that the substance is easily scraped or broken away from the drum. One means of applying the powder to the bare part of the drum between the knife and the feed bath comprises a long narrow jet as long as and close to the drum which is supplied by one fan with the air-borne dust. Another fan produces a suction through a pair of hoods closely surrounding the supply jet, through which any excess powder is withdrawn, collected in a cyclone, and re-used. B. M. VENABLES.

Mixing and emulsifying apparatus. A. W. EMPSON (B.P. 282,466, 26.6.26).—The apparatus comprises a centrifugal rotor with small labyrinthine passages through which the mixture passes. The outlets from the rotor are still smaller than the passages, so that

the passages are maintained full of mixture and the size of the outlets can be varied by changing grub screws or other means. A collecting casing is provided for excess feed as well as for emulsion, and in the latter are vanes for arresting the motion of the emulsion and aerating it. The amount of aeration is controlled by varying the amount of air allowed to be drawn into the emulsion casing from the excess feed casing.

B. M. VENABLES.

Concentration of liquids. A. HUIILLARD (F.P. 621,358, 7.1.26).—A liquid is heated to a temperature approaching that at which gas bubbles are formed, and the formation of these is limited to the surface of the liquid by maintaining the pressure at that exerted during the heating.

L. A. COLES.

Apparatus for expressing liquid constituents from materials of various kinds. F. KRUPP GRUSONWERK A.-G. (B.P. 273,689, 21.6.27. Ger., 29.6.26. Addn. to B.P. 248,372: B., 1927, 176).—The pressing finger and removing scraper of the device described in the original patent are interconnected so that when the latter moves downwards under increasing pressure of the material, the former moves in the same direction but to a (usually) much smaller extent. The actual travel, the travel ratio, and the spring load are all adjustable.

B. M. VENABLES.

Rotary water-screening plant. F. W. BRACKETT & Co., LTD., and F. W. BRACKETT (B.P. 282,569, 1.2.27).—A rotary cylindrical screen, *e.g.*, for condensing water, is rotated by the filtered water leaving the screen, acting on vanes round the periphery.

B. M. VENABLES.

Separators and filters for liquids. J. H. DINE and C. H. BILLINGTON (B.P. 282,542, 15.11.26).—A number of frames for filter bags are held in place simply by wedging action between an inclined surface on one wall of the tank and the apertured outlet channel along the other side of the tank.

B. M. VENABLES.

Apparatus for controlling chemical reactions. H. S. HATFIELD, and UNITED WATER SOFTENERS, LTD. (B.P. 282,168, 18.9.26).—An automatic machine is provided to analyse a liquid and then to control reactions in the bulk of the liquid in accordance with the analysis. A measured portion of the liquid is taken, and to it is added a reagent in portions at a time until a critical point is reached or over-run. The critical point may be indicated by change in electrical conductivity (or other physical properties) or, as in water testing, by the formation of froth. The number of portions of test reagent will control the addition of the bulk reagent.

B. M. VENABLES.

Means for effecting the chemical analysis of liquids and controlling chemical operations. II. S. HATFIELD (B.P. 282,170, 20.9.26).—A sample of liquid is subjected to a chemical reaction (*e.g.*, boiling to remove temporary hardness of water) and compared with an immediately following sample of the raw liquid by putting the two samples in the arms of a Wheatstone bridge. If the reaction is effected by electrolysis, the same electric current may be used in the bridge and to control, by electro-magnets, the measuring device by which the reaction is caused to regulate the addition of bulk reagent.

B. M. VENABLES.

Liquid and gas contact apparatus. W. J. MCGURTY, Assr. to BARTLETT HAYWARD Co. (U.S.P. 1,656,591, 17.1.28. Appl., 19.6.25).—A laterally-curved chamber in the upper part of a casing communicates at one end with the interior of the casing through a passage extending centrally thereof, and is connected at its other end to a discharge port. Spaced vertical plates are mounted in the chamber with spaced vertical wires between them.

H. HOLMES.

Treatment of gases or vapours with liquids or liquefiable mediums. E. M. SALERNI (B.P. 282,493, 21.9.26).—The liquid is picked up by a rotating spiral scroll, similar to a Frenier pump, except that there may be several concentric spirals. The gas is forced through the spirals from the axis outwards, *i.e.*, counter-current to the liquid. The pick-up openings at the ends of the spiral may be adjusted to vary the rate of treatment.

B. M. VENABLES.

[Air] classifiers. A. H. STEBBINS (U.S.P. 1,655,575-7, 10.1.28. Appl., [A] 24.11.26, [B] 15.12.26, and [C] 24.12.26).—Modifications of an air separator are claimed in which the material slides down a number of conical or V-shaped baffles, the spaces between which form settling chambers, the whole being enclosed by an outer shell. Air jets blow upwardly in (A), outwardly in (B) from a central supply pipe, or across from wall to wall in (C), and propel the material into the settling chambers, whence the fine material only is carried out by the air currents, aided if necessary by an outlet fan also.

B. M. VENABLES.

Chemical heat storage. F. W. STERLING and H. L. CROOKER, Assrs. to B. H. DYER Co. (U.S.P. 1,656,366, 17.1.28. Appl., 22.8.23. Renewed 8.4.27).—An unstable solution which evolves heat on crystallisation is used, which consists of sodium acetate, calcium chloride, and water, the water being 46% of the total.

H. ROYAL-DAWSON.

Refining and purifying of liquids. P. T. SHARPLES and L. D. JONES, Assrs. to SHARPLES SPECIALTY Co. (U.S.P. 1,657,941, 31.1.28. Appl., 2.4.24).—See B.P. 231,877; B., 1926, 1006.

[Continuous] charging of furnaces with solid material. POWER-GAS CORP., LTD., and N. E. RAMBUSH (B.P. 283,238, 4.8.26).

[Grooved] building block or brick for use in furnaces and other structures. H. S. WOOD, T. WILLIAMS, and SOUTH DURHAM STEEL & IRON Co., LTD. (B.P. 283,307, 30.10.26).

Device for mixing liquids in variable proportions. H. KAUWERTZ (B.P. 282,533, 24.9.26).

Continuous kilns (B.P. 283,009).—See VIII. **Concentration of materials** (U.S.P. 1,656,042).—See X. **Determination of gas in water** (B.P. 282,487).—See XXIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Nitrogenous matter in coal. C. W. SHACKLOCK and J. DRAKELEY (J.S.C.I., 1927, 46, 478-481 t).—The quantity of nitrogen extracted by means of dilute sulphuric acid from peat, lignite, sub-bituminous, bituminous, and anthracite coals decreases in the order

given, and is not influenced, under the conditions of the experiment, by particle size provided the coal is first passed through a 30-mesh sieve. Nitrogen in the form of amide compounds, monoamino- and diamino-acid has been distinguished and determined. The monoamino-acids appear to be the most persistent compounds, and in the case of coals account for over 70% of the extracted nitrogen. The diamino-acids exist in coals in amounts which are too small to determine by the methods employed in this investigation. The amide compounds are present in all the fuels, and account for 15–20% of the nitrogen extracted from the coals.

Present-day knowledge of the humus and bituminous constituents of coals. H. TROPSCH (Brennstoff-Chem., 1927, 8, 369–374).—A review of recent work shows that much is known regarding the humus substances in "humus coal" (clarain), but much less of the composition of the bitumens. The humus constituents of peat and the younger lignites, of the older lignites, and of coal are composed of free humic acids, humic acid anhydrides, and alkali-insoluble humins, respectively, all closely related. Oxidation, with air under pressure and in presence of sodium carbonate, of lignin, lignites, and coals gives rise to similar products (e.g., mellitic and benzenepentacarboxylic acids), indicating that the humus substances contain the benzene nucleus, whilst the oxidation of cellulose yields products such as fumaric and succinic acids. A similar conclusion regarding the humus substances may be drawn from the nature of their hydrogenation products, and the phenolic character of humic acids is suggested by their yielding nitrophenols with nitric acid. Conversely, humic acids can be synthesised from the higher phenols and their derivatives. For similar reasons the lignin molecule appears to be to a large extent aromatic and phenolic. Lignin can, moreover, be converted into humic acids, with accompanying loss of methoxyl, by both chemical and biological means. The bituminous portion of lignites contains resins and waxes, the latter being partly composed of esters (e.g., ceryl and myricyl) of normal fatty acids (e.g., the C₂₅, C₂₇, C₂₉, and C₃₁ acids). The bitumen of coal can be separated into oily and solid compounds, the former comprising a mixture of hydrocarbons, whilst the latter contain oxygenated substances.

W. T. K. BRAUNHOLTZ.

Method of testing the strength of blast-furnace coke. W. WOLF (Stahl u. Eisen, 1928, 48, 33–38).—An apparatus for testing the strength and suitability of a coke for blast-furnace use comprises a vertical, rectangular vessel, two opposite sides at the lower end of which are provided with hinged doors which may be kept open at any desired angle by means of weights which are so adjusted that they allow the doors to open further when the pressure of the coke exceeds 2.5 kg./cm.² The vessel is charged with $\frac{1}{3}$ m.³ of the coke to be tested and the doors are opened at an angle of 45°; pressure of 1–2.5 kg./cm.² (according as the coke is hard or soft) is applied by means of a plunger pressing on the top of the coke column, and the material passing through the doors is screened on a sieve with 30-mm. holes. The percentage of undersize is called the "pressure abrasion loss," and the values so obtained are a measure of the strength of the coke in the blast furnace. This

procedure affords a more satisfactory method of classifying cokes than does the method involving agitation of the coke in a rotating drum.

A. R. POWELL.

Chemistry of Japanese plants. IX. Studies on the "Tundra" (peat) of Southern Karafuto. S. KOMATSU and O. HUKI (Sexagint [Osaka Celebration], Kyoto, 1927, 229–240).—An examination of peat from different depths shows that as the depth and age increase, the resin, humic acid, and lignin contents rise whilst the sugar and cellulose contents fall. It is concluded that cellulose and other carbohydrates, as well as lignin, play an important part in the formation of coal. Active charcoal, prepared from the peat by washing, drying, carbonising at 500–600°, treating with 25% caustic soda solution, washing, and heating at 700–800° for 1 hr., compared favourably with "norit" in adsorptive power.

C. D. LANGFORD.

Production and composition of decolorising carbons. P. HÖNIG (Chem.-Ztg., 1928, 52, 7–8, 34–35).—The methods of production of the four main classes of absorbent carbons are: (A) carbonisation of raw materials derived from plants, e.g., Bragg's charcoal, Carbox, Suchar, etc.; (B) the action of activating or inflammable gases at a high temperature on carbonised materials, e.g., Norit, Eponit, Supranorit; (C) carbonisation of raw materials mixed with carbonate of calcium or magnesium, e.g., Darco; (D) dehydration of raw materials such as carbohydrates with zinc chloride, e.g., Carboraffin. The products usually contain varying amounts of moisture, ash, and hydrogen, with traces of sulphur and nitrogen. Those of class (B) are richest in carbon, Supranorit containing 98.2%, whilst their absorptive power varies inversely as the hydrogen content. In Darco the lime and magnesia are firmly combined and cannot be removed by washing with acid. Carboraffin may be regarded as a dehydrated hydrocarbon in colloidal dispersion, which is practically insoluble and owes its absorptive power to its highly unsaturated character.

F. R. ENNOS.

Application of distillation under partial pressure of substances of high mol. wt. by means of highly superheated steam in order to effect the "demolisation" of highly "comolised" substances. H. H. WISLICENUS (Z. angew. Chem., 1927, 40, 1500–1506).—[With H. LIANG.] Distillation with highly superheated steam may be carried out in the Schrader aluminium retort (B., 1921, 500 A), or in a specially constructed apparatus, in which superheated steam passes round and then through a central cylindrical retort, which may be placed horizontally or vertically, containing the substance. Highly superheated steam may cause a purely physical dispersion ("demolisation") of highly aggregated ("comolised") molecular systems, which cannot be distilled even under reduced pressure. In other cases it causes definite thermochemical decomposition or simple hydrolysis. When rosin is subjected to distillation with steam at 240–250°, the distillate (or sublimate), obtained in 86–96% yield, comprises pure, crystalline abietic acid, whilst a small quantity (about 0.5%) of carbon dioxide is simultaneously evolved. After recrystallisation from alcohol the acid melts at 154–162° (with selected crystals, 159–164°),

whilst on keeping, even in an evacuated desiccator, the crystals run together to a pale yellow resin. The mol. wt. of abietic acid obtained from commercial rosin, and from freshly collected fir resin, whilst giving the same ultimate analysis, was 308 and 517–518, respectively. The great readiness of abietic acid to “comolise” is evidenced by its separation in large white aggregates from the resin obtained from the living parts of fir trees. The distillation of dammar resin with superheated steam requires a higher temperature (340–350°) than rosin, as well as more steam and a longer distillation time. The distillate does not crystallise, but melts at about 50–80°.

[With E. STELZER.] With superheated steam amber yields non-crystallising oils, of which the viscosity and depth of colour increase the higher the temperature of the superheated steam used (up to 350°).

W. T. K. BRAUNHOLTZ.

Destructive distillation of wood and cellulose under pressure. K. FROLICH, H. B. SPALDING, and T. S. BACON (Ind. Eng. Chem., 1928, 20, 36–40).—The yield of methyl alcohol obtained by the destructive distillation of birch wood is increased by 65–80% over that obtained at atmospheric pressure when the operation is conducted under autogenous pressure at 280–370°. A further increase in temperature causes a considerable decrease in the yield owing to decomposition of the products initially formed. When distillation is carried out under a pressure of 200 atm. in a current of hydrogen, still higher yields (up to 3·11% on the weight of dry wood) are obtained. These increased yields of alcohol are not derived from the cellulose molecule, since only traces of methyl alcohol are formed when cellulose is distilled under the same conditions. Catalysts, in so far as these have been examined, usually reduce the yield of methyl alcohol at atmospheric pressure, and have but a slight effect at higher pressures, though in one case, with a copper-zinc-aluminium catalyst at a hydrogen pressure of 200 atm., 4·77% of methyl alcohol was obtained, possibly due to the fact that the catalyst was added as acetate. With nickel acetate, however, distillation under 200 atm. hydrogen pressure to a final temperature of 500° results in the conversion of more than 90% of the wood into volatile products, including phenols and saturated and unsaturated hydrocarbons. Similar products result when cellulose is substituted for wood; in this case over 97% of the cellulose is converted into volatile products, and even when hydrogen is replaced by nitrogen a conversion of 84% is realised.

D. J. NORMAN.

Hydrocarbon synthesis from carbon monoxide and hydrogen. O. C. ELVINS (J.S.C.I., 1927, 46, 473–478 T).—The interaction of carbon monoxide and hydrogen was investigated at atmospheric pressure and in presence of catalysts at temperatures of the order of 280°. A catalyst prepared from a mixture of 60 pts. of cobalt, 30 pts. of copper oxide, and 45 pts. of manganese oxide was used in conjunction with varying gas mixtures. Contrary to the original statements of Fischer and Tropsch that only aliphatic hydrocarbons are formed by this reaction, definite amounts of water-soluble oxygenated compounds and olefines were found among

the reaction products. A detailed account of the experimental work is given and the preparation of the catalyst from precipitated hydroxides, by ignition of nitrates, and from fused oxides, is described. The size of the granule was also investigated from the point of view of the inherent fouling effect of the waxy products of reaction.

Composition of cracked distillates. J. S. S. BRAME and T. G. HUNTER (J. Inst. Petrol. Tech., 1927, 13, 794–816).—Experiments have been made to distinguish between cracked spirit and straight-run petrol, quantitative information as to the constituents of which is very scanty owing to the difficulty of separation even into unsaturated, aromatic, naphthene, and paraffin hydrocarbons. Fractional extraction was first investigated, with methyl alcohol, sulphur dioxide in acetone, nitrobenzene, aniline, and dimethylaniline, the changes which occurred being traced by determination of the aniline dissolution point, but in no case was a satisfactory separation achieved. The dissolution points of a number of pure hydrocarbons with aniline and with nitrobenzene are also recorded. Attempts to separate unsaturated hydrocarbons, at first from mixtures of known composition, by means of mercury derivatives, proved unsatisfactory because the course of the reaction was uncertain and not quantitative; the solid products were also much contaminated with unchanged oil, and their reconversion into free hydrocarbons was difficult. Successful separation was finally accomplished by bromination followed by fractionation, and the hydrocarbons were recovered by the action of copper-zinc or aluminium-mercury couples. In dealing with the analysis of paraffins and naphthenes, mixtures of hexane and cyclohexane were used. It was found that the addition of a third liquid, followed by careful fractionation, made possible separation to a far greater degree than could be achieved by distillation of the binary mixture; the best results were obtained by the use of aniline. The foregoing methods were applied to a cracked spirit from Russian kerosene, boiling below 120°; this was divided into 11 fractions which were examined in detail, and many individual hydrocarbons were isolated and identified. It is concluded that both paraffins and isoparaffins are present accompanied by nearly equal quantities of naphthenes, and that olefines constitute the bulk of the lower-boiling unsaturated hydrocarbons, but diolefines are also present and cyclic olefines occur in the higher-boiling fractions; only small amounts of aromatic substances are present.

R. H. GRIFFITH.

Determination of nitrogen in petroleum and bitumens. E. J. POTH, W. D. ARMSTRONG, C. C. COGBURN, and J. R. BAILEY (Ind. Eng. Chem., 1928, 20, 83–85).—Determination of nitrogen in petroleum by the Kjeldahl method has been generally regarded as inaccurate, but the following procedure has been found reliable. The oil is digested with a large excess of sulphuric acid in the presence of potassium sulphate, mercuric oxide, and copper sulphate, and the resulting mixture is treated in the usual way, with precautions necessitated by the large amount of acid employed. Results obtained in this way with numerous oils are compared with those given by the combustion method,

and are considered to be more reliable; they also suggest that figures previously published for the nitrogen content of Californian petroleum are too high. R. H. GRIFFITH.

Effect of sulphur in gasoline on wrist-pin corrosion in automobiles. S. H. DIGGS (Ind. Eng. Chem., 1928, 20, 16—17).—Corrosion of automobile engines due to sulphur in the fuel is particularly noticeable in wrist-pins, but only occurs in cold weather. Tests were made, on an engine fitted with new pistons, rings, and wrist-pins, with three samples of gasoline containing 0.040, 0.151, and 0.458% S, respectively; with the first there was no perceptible corrosion, but with the second it was definitely present, and with the third very serious. Free sulphuric acid was detected in the crank-case, but no other acid was present. All forms of sulphur in the fuel are oxidised to sulphur dioxide, some of which will enter the crank-case and in the presence of water and air will form sulphuric acid, but in warm weather or in engines running continuously insufficient water will condense to make this possible. R. H. GRIFFITH.

Sulphur in gasoline from the automobile point of view. II. C. MOUGEY (Ind. Eng. Chem., 1928, 20, 18—21).—If the permissible sulphur content of gasoline could be raised from 0.1 to 0.3% great reductions of refining costs would result. Attempts have been made to render this possible by crank-case ventilation and control of water-jacket temperatures so that condensation of water is avoided; a large number of tests carried out on a variety of cars show that corrosion can be prevented in this way, but no recommendation is made to allow higher sulphur contents until automobile equipment of this type becomes general. R. H. GRIFFITH.

Preparation of fuel oil by dry distillation of the calcium soap of soya bean oil. IV. Comparison with the magnesium soap. M. SATO. V. Hydrogenation of the distilled oil. M. SATO and H. MATSUMOTO (J. Soc. Chem. Ind. Japan, 1927, 30, 242—245, 245—252).—IV. The use of the dry distillation products of the magnesium and calcium soaps of soya bean oil in the preparation of fuel oil has been investigated. The best result was obtained by heating either of the soaps at temperatures below 450° for several hours, followed by distillation above 500°. The time required for completing the distillation of the magnesium soap was less than that for the calcium soap at the same distillation temperature; similarly, the sp. gr. of the distillate of the magnesium soap was somewhat greater than that of the calcium soap. V. The light oil (100—175°) and middle oil (175—300°) were hydrogenated in liquid or vapour phase, using nickel reduced at about 370° as catalyst, and the products examined. The hydrogenation of the light oil began at about 60° and was rapid at 80°. In vapour phase it was readily reduced to saturated hydrocarbons at 190°, forming a colourless oil having no disagreeable odour. The middle oil could be hydrogenated in liquid phase at 140° with 2% of nickel, its iodine value being decreased to about 60; similar results were obtained in vapour phase at 320°. Heptane, octane, and nonane were isolated from the hydrogenated light oil. The existence of aromatic hydrocarbons, such as benzene, toluene, and xylene, could not be confirmed. From the hydrogenated middle oil were isolated those constituents which had the

b.p. and ultimate composition analogous to those of $C_{11}H_{24}$, $C_{13}H_{28}$, and $C_{14}H_{30}$, but the sp. gr. of each fraction was greater than that of the corresponding hydrocarbon; it is inferred that some hydrocarbons of the naphthene series accompany the paraffin hydrocarbons, which are the main constituents of the oil.

Y. TOMODA.

Preparation of fuel oil by the dry distillation of the calcium soap of soya bean oil. VI, VII. Reaction mechanism of thermal decomposition of the calcium and magnesium salts of some higher fatty acids. M. SATO, also M. SATO and C. ITO (J. Soc. Chem. Ind. Japan, 1927, 30, 252—260, 261—267).—VI. Calcium and magnesium stearates were heated at 450—480° in a partial vacuum (5—7 mm.). By the isolation of stearone from the distillates obtained, the formation of the ketone by the thermal decomposition of the stearates is confirmed. By similar experiments the formation of oleone from the corresponding oleates was confirmed. The decomposition temperatures of these salts were measured, the results being expressed as curves relating acetone-soluble matter (%) and temperatures. VII. Rates of thermal decomposition of calcium and magnesium stearates and oleates at 400° have been measured, and the results are expressed by graphs. Analysis of the gases formed by the decomposition of stearone and oleone at different temperatures showed that the rate of formation of carbon monoxide from the ketones was very small at 300—350°, and increased gradually with temperature. Thus, the carbon monoxide formed during 1 hr. from stearone and oleone heated at 500—550° and 550—600°, respectively, was 48.10% and 66.96% of the theoretical quantity. Saturated and unsaturated hydrocarbons and small quantities of hydrogen and carbon dioxide were also formed by the thermal decomposition of the ketones.

Y. NAGAI.

Constituents of petroleum from Maki, Echigo Province. S. IIMORI and U. KIKUCHI (J. Chem. Soc. Japan, 1927, 48, 520—526).—Petroleum from Maki was unsatisfactorily purified by means of concentrated sulphuric acid, from which a fraction (about 15% of the original oil) distilling at 125—155° was obtained. By bromination and nitration of the fraction, tetrabromoxylene, m.p. 250°, and trinitroxylene, m.p. 174°, were obtained. The bromination and chlorination of the same fraction from more satisfactorily purified oil gave tribromo- ψ -cumene, m.p. 234—237°, and trichloro- ψ -cumene, m.p. 215—216°. The presence of xylene (or a mixture of *m*- and *p*-isomerides) and hexahydro- ψ -cumene in the petroleum was therefore confirmed. The m.p. of tribromo- ψ -cumene was higher than that (222—224°) prepared from Merck's product. K. KASHIMA.

Micro-structure of New Zealand lignites. W. P. EVANS (Fuel, 1928, 7, 75—83).—See B., 1928, 3.

Potassium ferrocyanide. CHEMNITIUS.—See VII. Catalytic decomposition of soya bean oil. HAGA.—See XII.

PATENTS.

Treatment of carbonaceous materials. R. LESSING (B.P. 232,874, 27.9.26).—Coal or similar material is cleaned by gravity separation in a liquid of suitable

density, *e.g.*, an alkaline solution of calcium chloride. The coal is introduced below the surface of the separating liquid, which is agitated at the point of introduction. The "float" and "sink" are continuously removed separately, and are washed by upward or downward displacement of the bath liquor with a continuous unbroken body of washing liquid. It is advantageous to remove the greater part of the dust in the coal before submitting it to the process. In a modification of the process the washing may be incomplete and a predetermined quantity of the solute may be left in admixture with the coal to act as a catalyst during subsequent carbonisation, hydrogenation, etc. A. B. MANNING.

Manufacture of fuel briquettes. E. W. BOWEN (B.P. 282,890, 30.9.26).—The fine dust is first removed from the fuel to be briquetted, the remaining granular portion is mixed with the requisite quantity of binder, and then a definite proportion of the dust is re-introduced into the mixture, which is finally moulded and baked. Suitable proportions to use when briquetting anthracite duff are 86% of granular material, 9% of binder (waste sulphite liquor), and 5% of dust. The dust may be separated from the granular material in the drying chamber by an upwardly moving centrifugal draught. Addition of the binder and subsequently of the dust is carried out during the passage of the material through screw conveyors, in which the mass is effectively mixed.

A. B. MANNING.

Manufacture of highly active carbon blocks or granules. CHEM. WERKE CARBON GES.M.B.H. (B.P. 266,674, 29.11.26. Ger., 24.2.26).—Finely-divided carbon (1–10 μ in size) is mixed with an organic binding material, *e.g.*, 60–70 pts. of wood charcoal to 30–40 pts. of tar, containing a soluble inorganic substance such as caustic soda lye as a catalyst. The mixture is moulded under pressure (300 atm.), dried, carbonised, and activated in the usual way with gases or vapours. To accelerate the drying and oxidation process, oxides, resins, or linoleates of manganese, lead, cerium, or cobalt may be added. The inorganic substances are subsequently extracted from the finished product.

A. C. MONKHOUSE.

Manufacture of a highly active alkaline coke. N. V. NEDERLANDSCHE MIJNBOW EN HANDEL MAATSCHAPPIJ (B.P. 265,993, 11.2.27. Ger., 12.2.26).—Coal, peat, asphalt, or wood, etc., mixed with alkali or barium carbonates or hydroxides, together with a binder if necessary, and a catalyst, *e.g.*, an iron or nickel compound, if desired, is carbonised to produce a granular active coke suitable for the fixation of nitrogen in the form of cyanide. The finely-divided material is fed continuously on to the external surface of a drum heated internally and rotating inside a gas-tight chamber. Projecting ledges on the drum prevent the material from falling, until it is discharged at the bottom of the chamber, when, if required, it may pass to a second carbonising chamber at a higher temperature. The vapours from the chambers form on condensation a valuable acid-free tar.

A. B. MANNING.

Fuel. W. H. ROGERS (B.P. 280,349, 25.10.26).—Road sweepings or waste combustible materials are mixed with an inflammable liquid, *e.g.*, $\frac{1}{2}$ pint of petroleum to

1 cwt. of sweepings, and the mixture is then moistened with water. Clay or similar binder may be added and the material compressed into briquettes. [Stat. ref. to B.P. 230,252, 158,671, 105,340, and 27,334 of 1910.]

A. C. MONKHOUSE.

Retort furnaces for the distillation of coal and similar carbonaceous substances. LOW TEMPERATURE CARBONISATION, LTD., and C. H. PARKER (B.P. 281,348, 7.6.26).—The setting of vertical retorts has between each series in line a common combustion chamber in which transverse walls of chequer brickwork are built. These form chambers into which the heating gases from the combustion chamber pass into contact with the retort walls.

A. C. MONKHOUSE.

Distillation or coking of coal. A. GAERTNER (B.P. 281,110, 29.12.26).—Finely-divided coal is distilled by being injected with combustion gases into a furnace consisting of a horizontal drum or vertical chamber containing a bell-shaped deflector with a suitable system of baffles or electric dust separator. The distillation gases are burnt in a chamber surrounding the distillation chamber, and the cooler for the gases is used to preheat the powdered fuel. Catalysts may be used in the distilling chamber or injected in the form of finely-divided metal.

A. C. MONKHOUSE.

Distillation of fuel. E. ROSER (B.P. 283,420, 29.6.27).—Fuel is carbonised in chambers forming the periphery of a rotary drum, which is heated both internally and externally. The drum is rotated in stages, and during the stationary periods a plunger, operating at a suitable position in the setting, compresses the material in the chamber opposite to it. A compact coke is thereby produced.

A. B. MANNING.

Distillation of solid fuel in small pieces. E. HUREZ and P. P. VIRELY (F.P. 620,307, 18.8.26).—The fuel, broken up into small pieces of 10–120 mm. diam., slides over an inclined hearth.

L. A. COLES.

Treatment of coal gas etc. and production of ammonium sulphate. C. COOPER, F. B. HOLMES, and W. C. HOLMES & Co., LTD. (B.P. 281,474, 30.11.26).—The cooled gases obtained from the distillation or gasification of fuels are washed in the presence of carbon dioxide with a mixture of ammonia liquor and calcium sulphate. The liquor used is either the condensed liquor or is obtained from it by distillation and, if necessary, is freed from pyridine, tar acids, and cyanogen compounds. The gases after treatment pass through sulphuric acid, which is subsequently used in the treatment of the mixture of ammonia liquor and calcium sulphate after conversion into calcium carbonate and ammonium sulphate.

A. C. MONKHOUSE.

Manufacture of gas. H. NIELSEN and B. LAING (B.P. 281,105, 25.8.26. Addn. to B.P. 262,834; B., 1927, 273).—Coke produced by the carbonisation of fuels by internal heating up to 1200° is used for the manufacture of gas as described in the prior patent.

A. C. MONKHOUSE.

Washing, purifying, and cooling of producer gases. E. WARNANT (B.P. 282,851, 2.9.26).—A gas scrubber suitable for use on automobiles driven by producer gas comprises a vertical column of small cross-sectional area and considerable length, filled with coke or

metal turnings, down which the gas passes together with water in a fine state of subdivision, and a subsequent separator also filled with coke or turnings and containing water through which the gas is caused to bubble. The separator surrounds the base of the scrubber, the overflow being level with the top of the former. The water is circulated continuously through the scrubber and through a heat interchanger or radiator.

A. B. MANNING.

Means for rendering harmless the exhaust gases of internal-combustion engines. D. A. MACKINNON (B.P. 281,113, 4.1.27).—The exhaust gases from each engine cylinder pass through a separate chamber lined with copper and containing copper screens; a whirling motion is given to the gases by means of deflectors, and air is admitted by pump or fan. A conversion of the carbon monoxide in the gases into carbon dioxide takes place.

A. C. MONKHOUSE.

Heating and cracking of oils. T. E. ROBERTSON. From POWER SPECIALTY Co. (B.P. 283,032, 20.4.27).—Oil is heated to a cracking temperature by passage through tubes heated by the hot gases from a furnace, the rate of passage of oil being rapid enough to avoid a large portion of the oil-cracking effect which would result if the oil were heated to the same temperature by less rapid passage through less highly-heated tubes. The gases leave the oil-heating tube zone at a temperature a little below the maximum temperature at which they may safely be brought into contact with a tubular steam superheater (*i.e.*, gases at about 816° are utilised to superheat steam to about 482°), and the degree of superheating is controlled by the regulated injection of water into the steam. The final residual heat of the flue gases is used to preheat the oil to be treated. C. O. HARVEY.

Refining of petroleum. SHARPLES SPECIALTY Co., Assees. of L. D. JONES (B.P. 259,553, 24.9.26. U.S., 7.10.25).—Petroleum residues remaining after the removal by distillation of gas, naphtha, and burning oils are freed from "non-pressable wax" prior to refining for the removal of impurities (other than hard asphalt), as these impurities assist in the separation of the wax; *e.g.*, the residuum is diluted with naphtha, chilled, and centrifuged. Residua containing excessive quantities of impurities (*e.g.*, hard asphalt) other than those easily removed by clay treatment, cause trouble owing to inefficient removal of wax etc., and such oils are given a preliminary mild acid treatment. Residua containing more asphalt than can be economically removed by an acid treatment can be brought to a condition satisfactory for dewaxing by distilling off all the lubricating content without cracking; the beneficial impurities pass over with the last fractions of the oil, whereas the asphalt remains in the still. The oil should be steam-distilled up to a temperature of about 371°. Oils devoid of the necessary beneficial impurities may be blended with suitable oils prior to dewaxing. The dewaxed oil may be purified by freeing from naphtha, clay treatment, fractionation, etc. C. O. HARVEY.

Purification of hydrocarbons and the like by liquid sulphur dioxide. ALLGEM. GES. F. CHEM. IND. M.B.H. (B.P. 268,726, 6.11.26. Ger., 30.3.26).—A preliminary treatment with 10–25% of the total sulphurous

acid is made in order to remove water from the material. This extract is isolated, and the sulphurous acid recovered. The fraction from which the water is removed is treated with the remainder of the sulphurous acid as in the Edeleanu process. A. C. MONKHOUSE.

Distillation of hydrocarbons. W. M. DUNCAN (U.S.P. 1,654,797, 3.1.28. Appl., 14.7.24).—The hydrocarbons are distilled and cracked under pressures greater than 50 lb./sq. in., and the resulting vapours are passed through a chamber containing a solid hydrocarbon material, whereby the volatile constituents of the latter are removed. The container for the solid material is duplicated so that renewal of the spent material in one container may be carried out while that in the other container is undergoing treatment.

C. O. HARVEY.

Apparatus for treating hydrocarbons. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,655,600, 10.1.28. Appl., 28.6.20. Renewed 12.3.26).—A horizontal cylindrical still is provided with a centrally-disposed heating member from which heating gases pass, and around which an annular still section is rotatably mounted. The hydrocarbon oil to be distilled is contained between the rotatable portion and the outer wall of the still.

C. O. HARVEY.

Treatment of hydrocarbons. A. SCHWARZ, Assr. to COAL & OIL PRODUCTS CORP. (U.S.P. 1,655,741, 10.1.28. Appl., 8.5.23).—Predetermined proportions of saturated hydrocarbons of low b.p. and unsaturated hydrocarbons of high b.p. are vaporised and the vapours mixed intimately. After condensation, the uncondensed hydrocarbons are compressed and returned to the vapours that are being mixed. C. O. HARVEY.

Treatment [cracking] of hydrocarbon oils. E. R. WOLCOTT, Assr. to TEXAS Co. (U.S.P. 1,654,771, 3.1.28. Appl., 1.10.24).—Hydrocarbon oils are cracked by introducing them into a chamber along with a combustible mixture, firing the mixture and thereby subjecting the oil to a cracking temperature under a high pressure, and finally releasing the pressure to effect distillation of the products. C. O. HARVEY.

Cracking of hydrocarbon oils and manufacture of cyanides. J. C. CLANCY (U.S.P. 1,651,114, 29.11.27. Appl., 28.12.25).—In the process of U.S.P. 1,567,241 (B., 1926, 486) the carbonaceous material is replaced by hydrocarbon oils. T. S. WHEELER.

Coking of hydrocarbon oils. R. E. WILSON and H. V. ATWELL, Assrs. to STANDARD OIL Co. (U.S.P. 1,654,200—1, 27.12.27. Appl., [A] 26.11.24, [B] 21.1.27).—The oil is heated to temperatures above 482° on the surfaces of the nickeliferous metal walls of a chamber. Volatile products are withdrawn and scraping means are provided for removing the coke deposit.

C. O. HARVEY.

Reconditioning of crank-case oil. L. L. ODOM, Assr. to PURITY CHEMICAL Co. (U.S.P. 1,655,660, 10.1.28. Appl., 27.9.26).—Crank-case mineral lubricating oil is reconditioned by distilling off the low-boiling diluents and cooling while filtering the residual oil, agitating with sulphuric acid for about 30 min. and washing with water, agitating the washed oil at

a temperature of about 200° with sodium hydroxide solution for about 30 min. and again washing with water, heating the oil and evaporating off any intermingled water, and, lastly, agitating with diatomaceous earth and filtering. C. O. HARVEY.

Desulphurising petroleum and products derived therefrom. A. MAILHE (F.P. 611,890, 16.6.25).—The oil is heated with reducing agents, *e.g.*, with stannous or ferrous salts, or with tin or iron, or their alloys, in the presence of hydrochloric acid, in vessels constructed of material resistant to the action of acid vapours and hydrogen sulphide, and provided with condensing apparatus at the top. L. A. COLES.

Refining of oils. E. A. RUDIGIER, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,656,710, 17.1.28. Appl., 12.3.25).—An apparatus for dehydrating and cracking oils consists of a cracking coil whence the heated oil passes to a conversion chamber and thence to a vapour separator and heat interchanger. The oil to be treated passes through the heat interchanger to a dehydrating chamber and thence to the cracking coil.

C. O. HARVEY.

Counter-flow [oil-refining] still. F. E. GILMORE (U.S.P. 1,655,998, 10.1.28. Appl., 23.4.25).—Sets of horizontal heating pipes are spaced vertically within a shell of rectangular cross-section with baffling material between them. Hot gases are supplied to the lowest set and pass thence through the successive sets above it. Bubbler trays are arranged in the upper part of the shell with baffling material above them. An inlet for hot oil is provided between the trays, and above this an inlet, controlled thermostatically, for cold oil. An outlet for vapour is provided near the top and a float-controlled oil outlet near the bottom of the shell. H. HOLMES.

Purification of benzol, petrol, etc. K. COX and P. J. McDERMOTT, Assrs. to REFINERS, LTD. (U.S.P. 1,658,285, 7.2.28. Appl., 25.2.26. U.K., 12.1.26).—See B.P. 269,242; B., 1927, 436.

Machines for breaking coal or other friable material. B. NORTON (B.P. 283,835, 9.11.27).

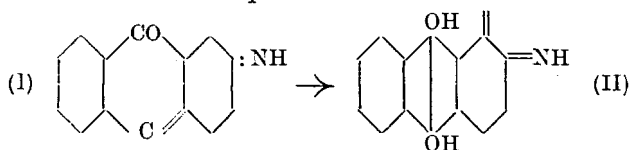
[Valve device for] supplying pulverulent fuels to internal-combustion engines. I. G. FARBENIND. A.-G. (B.P. 266,381, 21.2.27. Ger., 20.2.26).

Gas purifiers. H. H. HOLLIS (B.P. 283,283, 8.10.26).

Activated carbon (B.P. 283,267).—See VII.

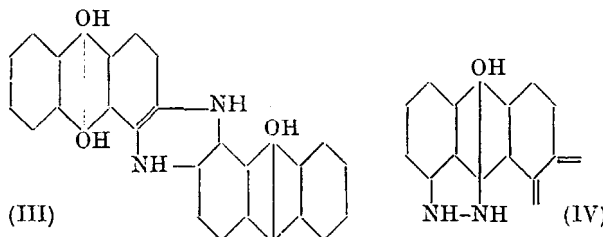
III.—ORGANIC INTERMEDIATES.

Alkaline fusion of anthraquinone derivatives. E. SCHWENK (Chem.-Ztg., 1928, 52, 45—46, 62).—Mechanisms for a number of potassium hydroxide fusion reactions in the anthraquinone series are based on the

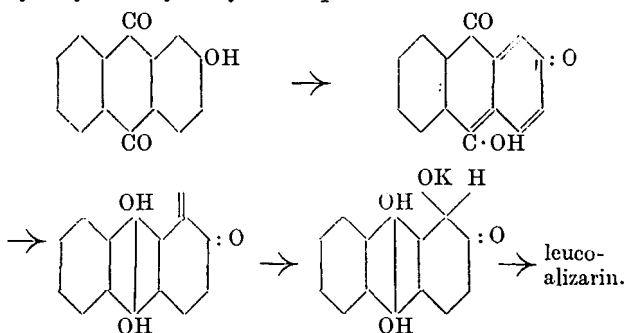


suggestion that one of the rings assumes a *p*-quinonoid configuration. The production of indanthrone, *e.g.*, is regarded as a simple condensation of 2 mols. of quinonoid 2-aminoanthraquinone (I, II). Below 200°

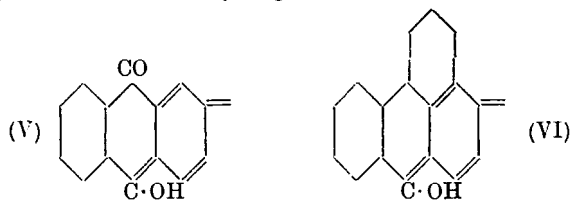
condensation is slow and oxidation to leuco-alizarin by way of 2-amino-1-hydroxydihydroanthraquinone is the main reaction. At higher temperatures tetrahydroindanthrone, accompanied above 300° by dihydroflavanthone, is formed. Since tetrahydroindanthrone undergoes oxidation-reduction with production of equal amounts of dihydroindanthrone and the useless trihydroxy-1 : 2 : 2' : 1'-dihydroanthrazine (III), the



yield of the former can never exceed 50% of the 2-aminoanthraquinone used. The conversion of pyrazolanthrone into Pyrazolanthrone Yellow may be figured similarly as the union of two *o*-quinonoid molecules (IV) to give first the tetrahydro-compound; and the yield of leucovat dye cannot exceed 50%. Alizarin formation from 2-chloro-, 2-bromo-, or 2-sulpho-anthraquinone proceeds by way of 2-hydroxyanthraquinone :



The low yield in absence of an oxidant is due to attack of leuco-alizarin by the hot alkali. A similar addition of K·OH to the *p*-quinonoid form (V) of anthraquinone accounts for the production of 2-hydroxyanthraquinone and leuco-alizarin by alkaline fusion of anthraquinone. Dibenzanthrone formation is figured as union of two *p*-quinonoid benzanthrone molecules (VI) followed by removal of two hydrogen atoms from the resulting



dihydrodibenzanthronyl, and a similar explanation is applied to the alkaline fusion of benzanthronequinoline, naphthalimide, etc. C. HOLLINS.

Activity of a nickel catalyst. E. J. LUSH (J.S.C.I., 1927, 46, 454—456 T).—In the hydrogenation of naphthalene with a nickel catalyst, it was shown that in the vapour phase only tetralin could be formed, whereas in the liquid phase decalin was produced. The total hydrogen absorbed was independent of the relative pro-

portion of tetralin and decalin made. Evidence was produced to show that in the hydrogenation of acetone, vegetable oils, and naphthalene, a given weight of activated nickel turnings used under standard conditions caused the same absorption of hydrogen per hour.

“Demolisation.” WISLICENUS.—See II.

PATENTS.

Laboratory apparatus for the production of absolute alcohol. ÉTABL. POULENC FRÈRES (F.P. 621,388, 12.1.26).—Several types of interchangeable glass dehydrating columns connected in series are attached to a central vessel, the whole apparatus being immersed in a water-bath.

L. A. COLES.

Manufacture of formaldehyde. SOC. CHIM. DE LA GRANDE-PAROISSE (AZOTE ET PROD. CHIM.) (B.P. 272,155, 7.9.26. Fr., 3.6.26).—Formaldehyde is formed by the interaction of carbon monoxide and water (liquid or vapour), thus: $2\text{CO} + \text{H}_2\text{O} = \text{CO}_2 + \text{CH}_2\text{O}$, in the presence of magnesium, zinc, cadmium, manganese, tin, lead, antimony, or bismuth, or their oxides or salts, alone or mixed, at 200–600°, preferably under pressure. The following metals and their oxides must be absent: chromium, molybdenum, tungsten, uranium, vanadium, niobium, copper, silver, gold, the iron group, and the alkali metals. The walls of the reaction vessel are made of inert metals (e.g., aluminium) or their alloys amongst themselves or with metals detrimental to the reaction (e.g., tinned copper). Thus, by using an aluminium tube, heated at 220°, containing manganese carbonate reduced with dry carbon monoxide at 150°, a dilute solution of formaldehyde was obtained, whereas with an aluminium-lined tube, at 340°, containing a mixture of magnesium oxide and antimony trioxide, a 12% solution was formed.

B. FULLMAN.

Method of condensation [of acetone]. N. W. THOMSON, ASSR. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,654,103, 27.12.27. Appl., 19.3.25).—Diacetone alcohol is obtained by leaving a suspension of potassium hydroxide in a mixture of acetone and benzene at room or lower temperature for 5–12 hrs.

T. S. WHEELER.

Manufacture of dinitrotoluene. E. H. RODD and R. W. EVERATT, ASSRS. to BRIT. DYE STUFFS CORP., LTD. (U.S.P. 1,657,723, 31.1.28. Appl., 8.12.26. U.K., 12.5.26).—See B.P. 263,018; B., 1926, 101.

Preparation of new derivatives of naphthoquinone. A. WAHL and R. LANTZ, ASSRS. to SOC. ANON. DES MAT. COL. ET PROD. CHIM. DE ST. DENIS (U.S.P. 1,657,809, 31.1.28. Appl., 12.7.24).—See B.P. 206,150; B., 1925, 200.

Production of α -aminoanthraquinones. J. THOMAS and A. H. DAVIES, ASSRS. to SCOTTISH DYES, LTD. (U.S.P. 1,657,420, 24.1.28. Appl., 20.7.21. U.K., 23.7.20).—See B.P. 173,006; B., 1922, 170 A.

IV.—DYE STUFFS.

Dyestuffs fast to ironing. III. R. HALLER, J. HACKL, and M. FRANKFURT (Kolloid-Z., 1928, 44, 83–84; cf. Haller, B., 1927, 840).—Solutions of dyes fast to ironing contain two or more disperse phases of different colours, which are in equilibrium with each other at a given

temperature. This was investigated by passing an electric current through the solution of the dye between two electrodes of white, semi-conducting material, such as filter paper. When Congorubin was treated in this way, after dialysis, using a difference of potential of 600 volts, the anode became blue, whilst the cathode remained white. With rising temperature, the anode became violet, later tending to scarlet. Addition of electrolytes such as sodium acetate, sodium chloride, ammonium chloride, and magnesium sulphate effected a reversal in the charge of one of the disperse phases, for, on electrolysis, the anode then became blue and the cathode red. Similar results were observed with Diamine Blue 3R, Bordeaux Extra, Benzazurin G and 5G, Benzo Brown RC, Brilliant Benzo Violet 2R, and Brilliant Benzo Fast Violet 2RL.

E. S. HEDGES.

Alkaline fusion of anthroquinone derivatives. SCHWENK.—See III. **Dyeing with sandal wood extract.** SOEP.—See XIX. **Light-sensitivity of dyes.** STEIGMANN.—See XXI.

PATENTS.

Manufacture of azo dyes [for wool]. I. G. FARBENIND A.-G. (B.P. 276,353, 19.8.27. Ger., 19.8.26).—2 : 5 : 6-Trichloro-*m*-toluidine-4-sulphonic acid (prepared by trichlorinating *p*-toluenesulphonic acid, nitrating, and reducing the product) is diazotised and coupled with a sulphonylpyrazolone, e.g., 1-(2-chloro-5-sulphophenyl)-3-methyl-5-pyrazolone, to give acid dyes fast to light.

C. HOLLINS.

Manufacture of new azo dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 282,151, 14.9.26).—Monoazo dyes, having as end-component a dihydroxynaphthalenesulphonic acid, are esterified with an arylsulphonyl halide in presence of an acid-binding agent. E.g., the dye *o*-anisidine \rightarrow 1 : 8-dihydroxynaphthalene-3 : 6-disulphonic acid, or aniline \rightarrow 1 : 6-dihydroxynaphthalene-3-sulphonic acid, is treated with *p*-toluenesulphonyl chloride. Good light- and fulling-fastness on wool is claimed.

C. HOLLINS.

Manufacture of [azo] dyes [pigments and ice-colours]. FABR. DE PROD. CHIM. ROHNER SOC. ANON. PRATTELN (B.P. 275,147, 6.12.26. Fr., 2.8.26).—Arylamides of 2 : 3-hydroxynaphthoic acid are coupled in substance or on the fibre with the tetrazo derivative of a diaminoazo compound obtained by coupling a diazotised mono-oxalyl derivative of benzidine, tolidine, dianisidine, etc. with a middle component and removing the oxalyl group by hydrolysis. Black pigments and dyeings result. An example is 4'-aminodiphenyl-4-oxamic acid \rightarrow α -naphthylamine, hydrolysed, tetrazotised, and coupled with 2 : 3-hydroxynaphthoic α -naphthylamide.

C. HOLLINS.

Manufacture of monoazo dyes capable of being chromed. I. G. FARBENIND. A.-G. (B.P. 263,816, 23.12.26. Ger., 23.12.25).—Chrome red dyes fast to fulling, potting, and light are obtained by coupling diazotised 5-nitro- or 4-chloro-5-nitro-*o*-aminophenol with 1-(2-hydroxy-5-sulpho-3-carboxyphenyl)-3-methyl-5-pyrazolone or the corresponding 3-carboxylic acid or ester.

C. HOLLINS.

Monoazo dyes. K. H. SAUNDERS and H. GOODWIN, ASSRS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,657,724, 31.1.28. Appl., 14.4.27. U.K., 17.4.26).—See B.P. 266,561; B., 1927, 325.

Manufacture of azo dyes. A. L. LASKA and A. ZITSCHER, ASSRS. to I. G. FARBEIND. A.-G. (U.S.P. 1,657,803, 31.1.28. Appl., 10.9.25. Ger., 20.9.24).—See G.P. 430,579; B., 1927, 213.

Inks (B.P. 279,963).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Action of chlorine and hypochlorous acid on wool. S. R. TROTMAN, E. R. TROTMAN, and J. BROWN (J.S.C.I., 1928, 47, 4—8 r).—The authors have shown (cf. B., 1926, 480) that the action of hypochlorous acid on wool is of a different nature from that of chlorine. Further investigation indicates that when chlorine reacts with wool soluble and insoluble chloroamines are formed, but none are produced by hypochlorous acid, although chlorine enters into combination with the wool proteins. Wool chlorinated with chlorine retains its power of condensing with semicarbazide, the increase in nitrogen content being the same as in the case of the untreated wool. On the other hand, after treatment with hypochlorous acid wool does not condense with semicarbazide. The condensation in the former case is not due to any reaction between the semicarbazide and the chloroamine group. Wool treated with chlorine in presence of sulphuric acid condenses with more semicarbazide than it does when chlorinated in presence of hydrochloric acid. Experiments were made with organic compounds containing CO and CO·NH groups. It is suggested that hypochlorous acid combines directly with the carbonyl groups of the protein molecule in one of the following ways. Direct combination might form $\cdot\text{NH}\cdot\text{C}(\text{OH})(\text{OCl})$ groups which if unstable would either give $\cdot\text{N}\cdot\text{COCl} + \text{H}_2\text{O}$, or the reaction may be $\cdot\text{CH}_2\cdot\text{CO} + \text{HOCl} = \cdot\text{CH}(\text{OCl})\cdot\text{CH}(\text{OH})$.

Action of acids on wool. S. R. TROTMAN, E. R. TROTMAN, and J. BROWN (J.S.C.I., 1928, 47, 45—49 r).—The rate at which dilute acids are taken up by wool was studied, the period of immersion being 24 hrs. With hydrochloric acid there was a regular increase in the amount of acid removed from the solution as the concentration increased from 0.01*M* to 1.0*M*. In the case of sulphuric acid, however, the maximum amount was taken up at 0.075*M*, and at 0.5*M* and 1.0*M* there actually appeared to be more acid present at the end of 24 hrs. than at the beginning of the experiment; similar results were obtained with acetic acid. These abnormal results are believed to be due to hydrogen sulphide or diffusible nitrogen compounds from the wool interfering with the titration, since normal results are obtained when the amount of sulphuric acid remaining is determined gravimetrically. It is suggested that the first stage of the action of acids on wool consists of sorption. More dilute solutions of sulphuric acid remove more nitrogen from wool than concentrated solutions. Probably, at high concentrations the surface film of sorbed acid becomes too viscous to allow diffusion of the products of reaction. Experiments on the effect of agitation on the rate of sorption seem to indicate that

the surface film is of considerable thickness. The effect of p_{H} on the action of acids on wool is considered and a scheme of equilibria is suggested. Experiments are described showing that hydrochloric acid prevents the condensation of semicarbazide with wool, but no such effect is observed in the case of sulphuric acid. Finally, some effects of acids on the physical properties of woollen yarns are described.

Degumming of silk. I. Action of alkali. II. Effect of formaldehyde in alkaline media. T. TAKAHASHI (J. Soc. Chem. Ind. Japan, 1927, 30, 568—573, 573—579).—I. The raw silk was treated with potassium hydroxide solutions of various strengths, and the amounts of sericin dissolved and alkali consumed were determined. It was observed that there are three stages in the action of alkali on sericin, viz., glutinisation (that is, swelling and the primary albuminate formation); dispersion; and secondary albuminate formation. The secondary albuminate thus formed stabilises the dispersed sericin particles and prevents them from being again adsorbed on the silk fibre. II. When formaldehyde was added to the degumming bath, or when the raw silk was treated with formaldehyde before degumming, the amount of sericin dissolved was less, and that of the consumed alkali more, than was the case when no formaldehyde was used. This is explained by assuming that formaldehyde lowers the dispersibility of the sericin and increases the reactivity of the latter towards alkali due to neutralisation of its amino-group. Y. TOMODA.

Constituents of the fibres of Japanese hemp-palm and sponge-gourd. S. MASUDA (J. Cellulose Inst., Tokyo, 1927, 3, 321—323).—The fibres, which are very resistant to decay in water, were purified mechanically, cut up, and analysed. The results (calculated on the dry material) for hemp-palm (*Trachycarpus excelsus*, Wendl.) and sponge-gourd (*Luffa cylindrica*, L., Roem), respectively, are: water, 11.34% (12.65%); benzene extract 0.77% (0.44%); alcohol extract 1.39% (2.9%); cellulose (on the extracted, dry substance) 42.6% (57.65%); reducible sugar, after hydrolysis of the isolated cellulose, 92.3% (99.7%) of the theoretical yield; lignin 37.5% (14%) with 0.54% (0.18%) of ash; and pentosan 16.8% (18.6%). Mucic acid was not isolated and only a trace of mannose-hydrazone was obtained from hemp-palm fibres, whilst those from sponge-gourd contained mannan 0.4%, galactan 1.8%. In both products, after hydrolysis with 3% sulphuric acid, xylose was identified.

B. P. RIDGE.

Rate of absorption of sodium hydroxide in the decomposition of straw at ordinary temperature. Y. KAUKO (Annal. Acad. Sci. Fennicæ [Komppa Jubilee Vol.], 1927, A29, No. 7. 30 pp.).—The rate of neutralisation of sodium hydroxide solution (6—12%) by straw at ordinary temperature is determined by measurements of conductivity. The velocity of reaction during the first stage is mainly controlled by the rate of diffusion into the tissue of the sodium hydroxide, which is neutralised in part by the silicic acid. This stage is followed by the dissolution of the lignins and the carbohydrates and the breaking up of the structure of the straw. G. A. C. GOUGH.

Pulping flax straw. III. Hydrolysis and delignification with sodium hydroxide and with a mixture of sodium hydroxide and sodium sulphide.

M. W. BRAY and C. PETERSON (Pulp and Paper Mag., 1928, 26, 37—41).—The results of a series of experimental cooks on flax straw at 155° using caustic soda alone and mixtures of caustic soda with sodium sulphide indicate that caustic soda-sodium sulphide liquors are more efficient than straight caustic soda liquors of the same available alkalinity (calculated as NaOH on the assumption that $\text{Na}_2\text{S} \rightarrow \text{NaOH} + \text{NaHS}$ by hydrolysis), in that the liquors containing sulphide remove lignin at a more rapid rate, and to a greater extent, whilst causing less destruction of the cellulose itself. For example, digestion for 7 hrs. with 15% of available alkali (13.4% of caustic soda and 3.3% of sodium sulphide) gives a pulp of approximately the same lignin content as is given by a straight caustic soda digestion for 10 hrs. with 20% of caustic soda. Further, chemical examination shows that the cellulose prepared with the sodium sulphide liquors is less degraded than that prepared with caustic soda alone. In comparison with sodium sulphite liquors, both caustic soda and caustic soda-sodium sulphide liquors are more rapid in their action, but cause a greater loss of cellulose for a given amount of lignin removed. D. J. NORMAN.

Sulphite pulp from spruce. D. E. CABLE (Pulp and Paper Mag., 1927, 25, 1215—1222, 1299—1305, 1331—1332, 1405—1408, 1422—1424).—Previous work dealing with the different phases of the sulphite process is reviewed and results are given showing the effects of various alterations in the cooking conditions in the experimental digestion of spruce wood. For spruce wood, though not necessarily for other woods, the pulp shows substantially the same degree of purity irrespective of the base (calcium, magnesium, ammonium, sodium) present in the cooking liquor. The injection of sulphur dioxide at the top of the digester during the first hour of the cook facilitates penetration of the chips by the liquor and reduces screenings (except in the case of sodium bisulphite liquors), with the result that increased yields of pulp of reduced bleach consumption are obtained. The effect of gradually lowering the digester pressure during the last few hours of the cook from the maximum (75 lb./sq. in.) to about 55—58 lb./sq. in., the liquor being maintained at about 148°, is to increase the yield of pulp, more particularly with straight calcium bisulphite liquors, by 1.2—2.7% above that obtained when the pressure is held at 75 lb. throughout the cook. This increased yield is, however, offset to some extent by an increased bleach consumption. D. J. NORMAN.

Sulphite-liquor lactones. B. HOLMBERG (Annal. Acad. Sci. Fennicæ [Komppa Jubilee Vol.], 1927, A29, No. 6. 16 pp.; cf. A., 1923, i, 1067; 1921, i, 772, 849, 850).—Sublimation of sulphite-liquor lactone, $[\alpha]_D - 55.2^\circ$ in acetone, at 225—260°/0.05 mm. gives rise to an isomeride, m.p. 206—209°, $[\alpha]_D + 30.7^\circ$, which is converted into β -dimethyl sulphite-liquor lactone by dimethyl sulphate. α -Dimethyl sulphite-liquor lactone is similarly converted into a mixture of almost equal parts of α - and β -dimethyl sulphite-liquor lactones. Treatment of either α - or β -dimethyl sulphite-liquor lactone with alcoholic sodium ethoxide leads to the formation

of a mixture of the two substances. The β -compound is changed to a dimorphous variety, m.p. 155.5—156.5°, by heating at 170°. α -Dimethyl sulphite-liquor lactone is oxidised by sodium hypobromite to oxalic acid; a neutral, optically inactive substance, $\text{C}_{20}\text{H}_{20}\text{O}_5$, m.p. 102—104°, containing 4 methoxy-groups; a dibasic acid, $\text{C}_{22}\text{H}_{24}\text{O}_8$, m.p. 193—194°, $[\alpha]_D + 36.9^\circ$ in acetone, containing 4 methoxy-groups (possibly tetramethyltruxillic or tetramethyltruxinic acid); and an optically inactive, monobasic acid, $\text{C}_{20}\text{H}_{20}\text{O}_6$, m.p. 224—225°.

G. A. C. GOUGH.

Pentosans in the sulphite[*-cellulose*] cooking process.

L. SHERJEBOW (Zellstoff u. Papier, 1928, 8, 87—88).—On heating with the cooking acid above 80°, pentosans form a compound with calcium bisulphite in which one pentosan molecule, consisting of four pentose residues, is united to one bisulphite molecule; increasing the temperature to 136° causes a further addition of bisulphite with the formation of a more stable compound in which the bisulphite molecule is united to two pentose residues. The fundamental unit of the pentosans $(\text{C}_5\text{H}_8\text{O}_4)_n$ may therefore be regarded as $(\text{C}_5\text{H}_8\text{O}_4)_4$, further hydrolysis causing a splitting of this into two parts in which $n = 2$. Above 137° rapid decomposition of this bisulphite-pentosan compound takes place with destruction of the pentoses, oxidation of the sulphur dioxide to trioxide, and the subsequent formation of calcium sulphate. If too little lime is present free sulphuric acid is formed. Sulphurous acid without bisulphite acts more quickly than bisulphite, but the reactions of the two are sharply distinguished from one another. If magnesium oxide is present instead of lime, the nature of the reaction between the pentosans and bisulphite is unaltered and a sulphur-magnesium compound remains at the end of the cooking. The formation of calcium sulphate is limited by the decomposition of the pentosan-bisulphite compound formed at the beginning of the reaction. The loose combination of bisulphite with the organic constituents of wood, which is destroyed by the action of ammonia with separation of calcium sulphite, or by heating with mineral acids with separation of sulphur dioxide, appears as a result of the addition of bisulphite to lignin or hexoses. The pentosans do not form such a compound.

B. P. RIDGE.

Preparation of cellulose nitrate. E. BERL and E. BERKENFELD (Z. angew. Chem., 1928, 41, 130—132; cf. Berl and Klays, B., 1907, 1255).—The nitrogen content and viscosity in acetone solution of a sample of cellulose nitrate is dependent rather on the composition of the waste nitrating acid than on that of the original acid mixture used, since an equilibrium is set up between cellulose nitrate and acid. Curves showing the nitrogen content and viscosity corresponding to varying waste acid compositions are given for both linters and wood cellulose, and the results are much more regular than those obtained when these properties are correlated with the composition of the starting acids. Increase of the sulphuric acid content above 60% causes a sharp diminution in the viscosity of the product, and the viscosity of nitrated linters is greater than that of nitrated wood cellulose with the same waste acid composition.

W. J. POWELL.

Effect of temperature on the rate of decomposition of nitrocellulose. R. W. RYAN and E. A. LANTZ (Ind. Eng. Chem., 1928, 20, 40—42).—The stability of nitrocelluloses may be measured by determining the rate of evolution of oxides of nitrogen at different temperatures and plotting the logarithm of the rate of evolution of nitric oxides, calculated as nitrogen, against the temperature. The resulting graphs are straight lines, and, when extended below the lowest temperature at which a test was made, give useful indications of the stability of nitrocelluloses at lower temperatures, whilst at the same time accentuating any differences between various samples. 2—5 g. of the nitrocellulose (previously air-dried and finally dried *in vacuo* over phosphorus pentoxide) are heated in a special decomposition tube provided with a spiral preheating coil for the air current, the whole being accommodated in a suitable vapour bath. Air, free from acid gases and moisture, is drawn through the sample at the rate of 4—5 litres/hr. and the oxides of nitrogen are absorbed in a cooled, neutral 5% solution of potassium iodide, the liberated iodine being titrated electrometrically. The time required to establish a constant rate of evolution of nitric oxides varies from 1—2 hrs. at 130—140° to 16—20 hrs. at 99—100°; titrations are therefore made at intervals of 30 min. at 137—140°, 1 hr. at 116—118°, and 3·5—4 hrs. at 99—100°.

D. J. NORMAN.

Viscosity of nitrocellulose. H. DABISCH (Farben-Ztg., 1928, 33, 1105—1106).—The statement by Merz (cf. B., 1927, 810) that the Engler viscosimeter gives "scientifically exact" measurements of the viscosity of nitrocellulose is criticised on the grounds that all nitrocellulose solutions are more or less plastic, but no distinction between plasticity and true viscosity is recorded in Engler determinations. An ideal viscosimeter will work at sufficiently high pressure to render plasticity effects negligible. From consideration of the influence of various solvents and diluents on the viscosity of nitrocellulose, a mixture of 45 pts. of absolute ethyl alcohol and 55 pts. of pure butyl acetate is recommended as the most desirable solvent. The concentration of the solution is adjusted for each type of nitrocellulose used so that normal lacquer consistency is obtained.

S. S. WOOLF.

Production of fibrous precipitates of substances and the structure of the fibres, particularly cellulose fibres. P. P. VON WEIMARN (Kolloid-Z., 1928, 44, 163—166).—General methods are described by which any insoluble substance can be precipitated in a fibrous form. In a double decomposition between two soluble salts, where one of the products is insoluble in the medium, by employing highly-concentrated solutions of the reactants the precipitate can be shown under the microscope to consist of thread-like structures. The threads are composed of a gelatinous sheath of the insoluble substance, containing a solution of one of the reactants. The further fate of the fibrous structure depends on the relative osmotic pressures of the solutions inside and outside the membrane. The structure of natural cellulose threads is very complicated, the fibres consisting of three concentric tubes enclosing layers of other substances. The outer sheath consists

of a hard skin of waxy material, the middle tube consists mainly of cellulose, and the inner tube of plasma. Between the first and second tubes is a layer of "hide"-cellulose, and between the second and third a layer of "plasma"-cellulose. The middle tube is in itself a complicated system of concentric fine tubes, the walls of which consist of ultramicroscopic fibres of cellulose. The chemical properties of the first and second tubes of wood cellulose are not identical with those of cotton cellulose, a marked difference being observable in the resistance towards the dispersing effect of concentrated solutions of very soluble salts. The high resistance of the outer sheath of cotton cellulose gives rise to an irregular swelling, which gives a characteristic appearance under the microscope.

E. S. HEDGES.

Change of plasticity of viscose with ripening. K. ATSUKI, T. TAKAGI, and T. OHTA (J. Cellulose Inst., Tokyo, 1927, 3, 317—320).—Measurements of the rate of flow of a viscose containing 7% of cellulose and 7·78% of sodium hydroxide, carried out in a modified Ostwald viscosimeter at 25° under pressures of from 3 to 25 cm. of mercury, after ripening the material at 25° for various times from 53 to 240 hrs., show that viscose has a slight but measurable plasticity, since the flow rate—pressure curve at a lower pressure is convex to the pressure axis, giving a yield value. As ripening proceeds, the yield value becomes smaller, reaches a minimum at 121 hrs., and again increases. It is supposed that the disperse phase, at first a continuous system, becomes less continuous by dehydration with the ripening, but that after maximum dehydration, when the yield value is a minimum, it begins to coagulate, forming a firm continuous system giving a yield value. The disperse phase, before and after minimum plasticity is reached, is more or less continuous, but differs in rigidity. In the former case it is easily deformable through being highly hydrated, whilst in the latter it is rigid on account of the formation of micelles of highly hydrated particles.

B. P. RIDGE.

Measurement of hydrogen-ion concentration in the control of pulp and paper manufacture. K. W. FRANKE and J. J. WILLAMAN (Ind. Eng. Chem., 1928, 20, 87—95).—From a review of methods of measuring the hydrogen-ion concentration it is concluded that the hydrogen electrode could be used for control purposes in the preparation of sulphite liquors providing that sulphitation is not carried beyond neutrality; below p_H 7 the readings are unreliable. The quinhydrone electrode is, in general, unsuited for the control of pulp and paper manufacturing operations. The most satisfactory results are obtained with the antimony electrode using ordinary pure stick antimony of radially crystalline structure. Both oxygen and carbon dioxide affect the potential of an antimony electrode, but, if partially closed electrode vessels are used and stirring is accomplished without unnecessary agitation, the results are trustworthy when the readings are taken within 2 min. Further, since all solutions contain oxygen, a tarnished electrode, though somewhat slower in response than a bright electrode, is more dependable. The antimony electrode correctly indicates the progress of the reaction in the preparation of neutral sulphite liquor from

alkaline leach liquors, and, being unaffected by sulphur compounds, is also suitable for determining the p_H value of the spent cooking liquor. For alkaline solutions, its upper limit of usefulness is about p_H 12. Other operations which could with advantage be controlled by the antimony electrode are engine sizing and water purification. The following equation is given for converting antimony electrode potentials into p_H values: $E = 0.050 + 0.054p_H$, at 25° (cf. Kolthoff and Hartong, B., 1925, 154).
D. J. NORMAN.

Economy of the sulphite-cellulose cooking process. A. FROBERG (Zellstoff u. Papier, 1928, 8, 83—87).—See B., 1928, 120.

Action of nitrous acid on wool. WATERMAN and GROOT. Mercerisation. LOTTERMOSER and RADESTOCK. —See VI.

PATENTS.

Treatment of hemp and other vegetable fibres. L. LAPIERRE (B.P. 279,946, 5.8.26).—The fibres in the form of a mat are placed on rotating screens in an autoclave, in which, after complete degumming, they are treated at 120° for about 2 hrs. with a solution consisting of potassium nitrate 10 kg., ammoniacal potassium sulphide 5 kg., sodium chloride 0.5 kg., sodium borate 0.5 kg., magnesium sulphide 1 kg., ammoniacal copper oxide 0.25 kg., and water 600 litres. The boiled fibre is washed, treated with 2% hydrochloric acid, and again washed until free from acid. "Oxaline oil" (1%) is then applied and the fibre passed through a 1% solution of potassium nitrate. The fibres thus acquire a silky appearance.
D. J. NORMAN.

Preparation of a wool-like material from jute, *Corchorus capsularis*, etc. P. GELLI (F.P. 613,973, 2.4.26).—The fibres are freed from impurities, opened up, and treated with caustic soda solution (d 1.263) at 25 – 30° . The mixture gradually becomes hotter of its own accord, and, when steam begins to appear, the fibres are removed from the bath, washed, and bleached.
D. J. NORMAN.

Washing or cleaning of wool. E. C. DUHAMEL, and COMP. GÉN. DES IND. TEXTILES (B.P. 251,669, 3.5.26 Fr., 4.5.25).—The process differs from that of B.P. 273,755 (B., 1928, 11) in that a substantially high but not necessarily constant concentration of suint liquor is maintained in one or more bowls of a washing set of the periodical-stoppage type, whilst a minimum quantity of fresh liquid (water or weak suint liquor), not substantially exceeding 1 litre per kg. of wool treated, is introduced into the installation. Methods are described for diminishing the losses of suint liquor and for purifying it for re-use.
D. J. NORMAN.

Treatment of fibrous filling material for use in the manufacture of plastic masses. K. RÖMMLER (G.P. 445,535, 31.3.25).—Cotton waste etc. is scoured with a degreasing agent, such as hot sodium silicate solution, after which it is dried, cut up into small pieces, washed to remove residual sodium silicate, and again dried.
L. A. COLES.

Preparation of artificial silk. T. IWASAKI (F.P. 621,543, 15.9.26).—Artificial silk of improved internal structure, i.e., with the particles arranged substantially

in the form of parallel chains, is obtained by converting viscose or similar colloidal silk-forming solution into a linear sol prior to spinning. This is effected by passing the solution through the field of a high-frequency electric current in a vacuum or in an atmosphere of an inert gas such as hydrogen. The solution is then ripened and stretch-spun.
D. J. NORMAN.

Production of high α -cellulose fibre. G. A. RICHTER, Assr. to BROWN CO. (U.S.P. 1,654,603, 3.1.28. Appl., 12.5.25. Renewed 3.6.27).—Sulphite pulp made with a liquor in which soda is the base is washed and digested with an alkaline liquor containing sodium compounds. The resulting high α -cellulose pulp is washed and a portion of these washings used for the alkaline digestion of further quantities of sulphite pulp, whilst the remainder is mixed with the washings from the sulphite process. This mixed wash-liquor is then treated for the recovery of sodium compounds.
D. J. NORMAN.

Manufacture of fine viscose filaments. VEREIN. GLANZSTOFF-FABR., A.-G. (G.P. 444,113, 21.5.19).—By using spinning baths containing considerably more acid than is customary in conjunction with viscose solutions of a suitable degree of ripeness, filaments of any desired denier may be spun through apertures of the usual size, e.g., 0.1 mm., by adjusting the concentration of acid according to the particular denier required.
D. J. NORMAN.

Hydrolysis of cellulose whereby high yields are obtained of products suitable for pure or symbiotic butyric fermentation. SOC. DES BREVETS ÉTRANGERS LEFRANC & CIE. (F.P. 616,424, 20.5.26).—Cellulosic material, e.g., wood, is partially hydrolysed by the action of sulphuric acid or sodium acid sulphate under such conditions of concentration and temperature that the products of hydrolysis are not destroyed. Hydrolysis is then completed by the action of hydrofluoric acid under pressure, the hydrofluoric acid being obtained by the addition of the calculated quantity of, e.g., calcium fluoride. The resulting liquor is neutralised with chalk and the precipitated calcium fluoride recovered.
D. J. NORMAN.

Apparatus for the dehydration of viscose products. S. A. NEIDICH (U.S.P. 1,654,553, 3.1.28. Appl., 1.5.26).—Wet viscose products are conveyed through a tubular conduit heated at the middle and cooled at the ends. A volatile liquid dehydrating agent is introduced into the conduit at the heated part and is recovered by condensation at the ends.
D. J. NORMAN.

Conversion of cellulose derivatives into a finely powdered form. LONARIT-GES. (G.P. 445,308, 7.7.23).—Plastic material containing, e.g., cellulose derivatives, camphor substitutes, and fillers, heated to render it sufficiently fluid, is forced through fine jets, and as it issues from these the solvent evaporates rapidly, causing the material to swell into porous threads which, after drying, are readily reducible to a fine powder.
L. A. COLES.

Manufacture of sheets, pellicules, or films of cellulose esters and ethers. H. J. HANDS, and SPICERS, LTD. (B.P. 281,803, 24.9.26).—A means is described for producing films etc. exhibiting a progressive variation

in constitution, or in mechanical or physical properties between their opposite faces. The compositions are applied in succession to a support, from which they are finally stripped, in substantially immediate succession, a second composition being applied before any material portion of the solvent has evaporated from the composition first applied.

W. CLARK.

Production of coloured material from cellulose esters. SOC. CHIM. DES USINES DU RHÔNE (F.P. 619,764, 3.8.26).—The colouring matter is formed in a solution of the cellulose ester in a suitable solvent, which is subsequently removed by evaporation; *e.g.*, a blue product is obtained by adding potassium ferrocyanide solution to a well-stirred mixture of cellulose acetate dissolved in acetone with ferric chloride solution, whilst a yellow product is obtained by using cadmium sulphate and sodium sulphide.

L. A. COLES.

Colloidising agent for nitrocellulose. T. L. DAVIS (U.S.P. 1,654,114, 27.12.27. Appl., 25.4.22).—The use of *s*-dialkylcarbamides is claimed.

T. S. WHEELER.

Recovery of by-products from sulphite-cellulose waste-liquor. L. PETIT-DEVAUCELLE (F.P. 619,861, 14.12.25).—The liquor is treated with alkali to convert the bisulphite into sulphite and dried to a moisture content of about 10%; 2–10% of gas tar is then added and the mixture completely dried to give a pitch-like product suitable for, *e.g.*, roads. Alternatively, part of the sulphur may be recovered by treating the liquor with hydrogen sulphide, after which air is passed in to convert calcium sulphite into sulphate whereby resins are separated and recovered. Resins may also be separated by treating the heated liquor with crude salt. The resins thus obtained are insolubilised by fusion and polymerisation, optionally after the addition of small quantities of natural resins, or they may be purified by the electrolytic hydrogenation of their solutions in soda.

D. J. NORMAN.

Treatment of the black liquor obtained in the manufacture of wood fibre by the soda process. E. HÄGGLUND (B.P. 273,267, 31.5.27. Ger., 23.6.26. Addn. to B.P. 258,035; B., 1927, 165).—The sodium acetate recovered by crystallisation, when the liquor discharged from the high-pressure tank is treated with a caustic alkali and concentrated, as described in the main patent, is contaminated with other soda salts. In order to avoid this, the liquor from the high-pressure tank is evaporated to dryness, without the addition of caustic alkali, and the sodium acetate recovered by fractional lixiviation of the dry residue, either before or after carbonisation at, *e.g.*, 300° to decompose other organic compounds such as formates, propionates, and butyrates.

D. J. NORMAN.

Separation of copper sludge from the coagulating liquors used in the manufacture of artificial silk. HÖLKENSEIDE G.M.B.H. (G.P. 443,089, 29.6.25).—The liquor is allowed to settle in large vessels with porous walls provided at different levels with valves for withdrawing the clarified liquor and near the bottom with a larger outlet for the sludge. As the clear liquor is removed, partly by decantation and partly by percolation through the porous walls, the sludge residue contracts and detaches itself from the sides of the vessel of its own

accord. Provision is made for mechanically removing the solidified sludge.

D. J. NORMAN.

Interrelated pulp refining and viscose process. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,653,124, 20.12.27. Appl., 29.10.25).—The pulp is purified with caustic soda solution, washed, formed into sheets, and converted into alkali-cellulose. The excess of soda removed from the alkali-cellulose by pressure is used for the preliminary treatment of further quantities of pulp.

D. J. NORMAN.

Production of waterproof paper or paperboard. L. MELLERSH-JACKSON. From BENNETT, INC. (B.P. 280,083, 28.2.27).—A paper furnish comprising waste wax papers alone or in admixture with other paper-making material is beaten at a sufficiently high temperature (about 77°) to soften or melt waxes or any other waterproofing agents, *e.g.*, rosin, which it is desired to incorporate therewith. 5–20% of rosin is then added and dispersed throughout the furnish, after which sodium silicate (*d* 1.41), in the proportion of 3–4 pts. per 1 pt. of rosin and preferably heated at 99°, is incorporated. When dispersion of the wax and rosin is complete the furnish is cooled to 49°, treated with alum until a faint acid reaction is obtained, and run on a paper machine. The resulting sheets are dried and heated to cause the wax and rosin particles to coalesce.

D. J. NORMAN.

Manufacture of a textile material from vegetable raw material. J. C. M. VAN DIGGELEN, Assr. to N.V. OCTROOI MAATSCHAPPIJ "VEDE" (U.S.P. 1,653,138, 20.12.27. Appl., 11.10.26. Holl., 29.4.24).—See B.P. 274,331; B., 1927, 774.

Manufacture of cellulose solution. Manufacture of viscose. L. LILIENTELD (U.S.P. 1,658,606—7, 7.2.28. Appl., 18.6.23. Austr., 15.3.23. Renewed [B] 23.11.27).—See B.P. 212,864—5; B., 1925, 37, 126.

Manufacture of artificial silk. F. W. SCHUBERT, Assr. to BRYSIKA, LTD. (U.S.P. 1,657,697, 31.1.28. Appl., 21.6.26. U.K., 2.7.26).—See B.P. 258,371; B., 1926, 1009.

Manufacture of hollow artificial silk threads. ERSTE BÖHMISCHE KUNSTSEIDEFABR. A.-G. (F.P. 621,181, 8.9.26. Czechoslov., 17.9.25).—See B.P. 258,582; B., 1927, 774.

Method and apparatus for obtaining fibrous material from stalks. E. GMINDER (B.P. 283,370, 17.3.27).

Photographic supports (B.P. 282,980).—See XXI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Action of nitrous acid on wool fibre and reactions of the product with azo components. H. I. WATERMAN and J. GROOT (Chem. Weekblad, 1928, 25, 18—22).—The production of colouring matter by treatment of wool fibre with nitrous acid and coupling appears to be due to adsorption rather than to true diazotation. There is no diminution of the nitrogen content of the fibre when it is treated with nitrous acid and boiled; the loss of nitrogen, which must therefore come from nitrous acid held up in some way in the fibre, is about 0.2% on boiling, but the treated fibre will take up considerably more H-acid than is equivalent to this.

The power of forming colouring matter with the coupling substances, also, is not lost even after the fibre has been treated with nitrous acid and then boiled several times. The treated fibre loses the power to become luminous under the influence of ultra-violet rays. S. I. LEVY.

Addition of pyridine to dye-vats. M. FREIBERGER (Z. angew. Chem., 1928, 41, 133; cf. Binz and Prange; B., 1928, 83).—The author refers to his previous publication (B. 1921, 175 A), not cited by Binz and Prange, in which the improvement in the rate of reduction of indigo obtained by the addition of pyridine and similar bases to the vat is described. W. J. POWELL.

Mergerisation of commercial cellulose and cellulose viscose. A. LOTTERMOSER and H. RADESTOCK (Z. angew. Chem., 1927, 40, 1506—1511).—The loss in weight and degree of swelling of various bleached and unbleached celluloses of different degrees of fineness, when mercerised with lithium, sodium, or potassium hydroxides, are determined and plotted graphically. With sodium hydroxide of increasing concentration the loss in weight rises, in every case, to a maximum at a concentration of 3.2 mols./litre, but the value of this mercerisation loss is least for the cellulose which has been most carefully treated in preparation (e.g., unbleached and coarsely ground). The loss increases with the time of steeping up to 12—16 hrs., then remains constant for about 8 hrs., and finally falls to about its initial value after 20—24 hrs. The loss at 100° is not appreciably greater than twice that at 20°. A specially designed volumenometer, containing mercury, for determining the degree of swelling of cellulose on mercerisation is described. Of the three alkalis examined, lithium hydroxide gives the greatest degree of swelling, and potassium hydroxide the least, but the curves drawn with molecular concentration of alkali as abscissæ and degree of swelling as ordinates are different in the three cases. With lithium hydroxide the curve has a maximum at a concentration of 2.8 mols./litre, with a horizontal portion on each side; with sodium hydroxide the maximum is at 3.15 mols./litre, and is followed by two horizontal portions; and with potassium hydroxide the maximum is at 3.8 mols./litre, and is followed by one long horizontal portion. The curve connecting time of steeping and degree of swelling is identical in appearance with the loss in weight-time curve. The curves showing the quantity of alkali taken up by cellulose from solutions of different strengths are not characteristic adsorption curves, and it is probable that true additive compounds are formed. This view is supported by the observation that these latter curves have horizontal portions at exactly the same molecular concentrations of alkali as are found in the curves for the degree of swelling. W. T. K. BRAUNHOLTZ.

Dyestuffs fast to ironing. HALLER and others.—See IV.

PATENTS.

Bleaching of vegetable fibres. R. VIDAL (F.P. 617,271, 22.10.25).—The moist material is treated, with or without previous digestion, first with gaseous chlorine in the absence of air, and then with a hypochlorite solution. D. J. NORMAN.

Dyeing, printing, or stencilling of materials made with or containing cellulose acetate. BRIT. CELANESE, LTD., G. H. ELLIS, H. C. OLPIN, and W. B. MILLER (B.P. 283,253, 30.9.26).—Acetate silk is dyed (etc.) with nitro-derivatives of compounds containing two or more aromatic residues, two of which are united by a single linking not consisting of nitrogen alone, i.e., excluding diphenylamines, azo dyes, and hydrazo compounds. Examples are 3:3'-dinitrobenzidine, 2-nitro-, 2:4-nitro-, and 4-chloro-2-nitro-*N*-benzylanilines, 3-nitro-4-aminodiphenyl ether, 3:3'-dinitro-4:4'-diaminodiphenylmethane, 3:3'-dinitro-4:4'-tetraethyl-diaminobenzophenone. Various shades of yellow are obtained. C. HOLLINS.

Application of azo dyes [to regenerated cellulose silks]. BRIT. DYESTUFFS CORP., LTD., and J. BADDILEY (B.P. 283,319, 10.12.26).—Level blue shades on viscose are obtained by the use of disazo dyes from *p*-nitro-aniline-*o*-sulphonic acid as first component, any suitable middle component, and as end component 2-amino-5-naphthol-7-sulphonic acid or its *N*-alkyl, aryl, or acyl derivatives. C. HOLLINS.

Fixation of dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 283,281, 7.10.26).—Acid or basic (triarylmethane) dyes are dyed on cotton, acetate silk, etc. previously mordanted with phosphotungstomolybdic acid, phosphotungstic acid, phosphomolybdic acid, or other derivatives of tungstic or molybdic acid. The mordant is itself fixed on the fibre by means of a preliminary mordanting with a fixing agent other than tannin (e.g., benzidine, phenol, tetramethyldiaminobenzylhydrosulphonic acid, dibenzylmetanilic acid, starch, sucrose, dextrose, lactose, cyclohexanol, etc.), and/or subsequent reduction with bisulphite, hyposulphite, formaldehyde, etc. C. HOLLINS.

Production of colour effects on fabrics. F. EDLINGER (F.P. 619,007, 16.7.26. Anstr., 20.4.26).—Fabrics, particularly cotton fabrics, are moistened and passed between hot rollers engraved with the required design. On subsequent dyeing, those parts of the fabric which have been pressed dye to a lighter shade than those that have not been so treated. D. J. NORMAN.

Increasing the wetting capacity of solutions. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H., G. BASEL, and F. KAUFLE (G.P. 444,966, 20.11.25).—An aliphatic amine such as butylamine, dibutylamine, diisobutylamine, or isoamylamine is used as a wetting agent, e.g., in dyeing with azo dyes. D. J. NORMAN.

Bleaching with hypochlorites. H. WENZL, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,657,140, 24.1.28. Appl., 23.6.26. Ger., 27.7.25).—See B.P. 260,190; B., 1926, 1011.

Dyeing of artificial silk. C. S. BEDFORD (U.S.P. 1,657,656, 31.1.28. Appl., 31.8.26. U.K., 21.9.25).—See B.P. 263,222; B., 1927, 186.

Dyeing of cellulose acetate products in black shades. A. J. HALL, ASSR. to SILVER SPRINGS BLEACHING & DYEING CO., LTD. (U.S.P. 1,657,255, 24.1.28. Appl., 10.6.26. U.K., 10.7.25).—See B.P. 258,699; B., 1926, 976.

Azo dyes (B.P. 275,147).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Mechanical salt-cake furnaces with indirect heat. The Mannheim furnace. F. BÖHM (Chem.-Ztg., 1928, 52, 101—102).—The Mannheim furnace was introduced about 30 years ago to work with the then abundant nitre-cake. This product was later replaced by sulphuric acid. It effected great economy in fuel and labour over hand furnaces. The nitre-cake was crushed and mixed with rock salt and delivered to the kiln by a screw conveyor. The kiln was fired by producer gas, and a charge required 5—6 hrs. to work, the usual output being 7 tons of salt-cake per 24 hrs., with a fuel consumption of 11—12%. Corrosion of the iron muffle was minimised by the use of dry nitre-cake and by the temperature being maintained at 850° or less. The quality of the iron is important. Parts of the stirring mechanism which were subject to severe corrosion were protected by ceramic or silicon plates. The life of the muffle was an output of 4500—5000 tons, but repairs were needed after an output of 2000 tons. The temperature necessary for calcination of the salt-cake approaches that at which corrosion by hydrochloric acid becomes severe. For working with sulphuric acid a higher temperature is required, and a ceramic muffle replaces iron. Salt and sulphuric acid are introduced separately in various ways, as external mixing gives troublesome incrustations in the feed pipe. The fuel consumption here is 18%. With both types of furnaces the waste heat is used for preheating the secondary air. C. IRWIN.

Manufacture of potassium ferrocyanide. F. CHEMNITZ (Chem.-Ztg., 1928, 52, 90—91).—Spent oxide from gas purification is ground, extracted with carbon disulphide to remove sulphur, and then washed with water to free it from ammonium compounds. The residue is then treated on a stirring vessel with sodium carbonate, calcium hydroxide, and water in quantities required by its content of "blue." The whole is stirred with air agitation for 3 hrs., tested, and, if decomposition is complete, put through a washing filter press. The filtrate passes into iron tanks. Here, if the calcium content is high it is reduced by treatment with mother-liquor from ferrocyanide crystallisation, and hydrogen sulphide, if present, is removed by blowing. It is then treated in wooden tanks with ferrous chloride, which is preferable to the sulphate. The precipitate of $\text{Na}_2\text{Fe}[\text{Fe}(\text{CN})_6]$ is filter-pressed, washed, and treated with lime and potassium carbonate in a stirring vessel with a heating coil, the quantities again being determined by test. The filtrate from pressing is evaporated in lead-lined pans and crystallised. C. IRWIN.

Preparation of finely-divided zinc oxide for ointments. E. DÖRZBACH (Pharm. Ztg., 1928, 73, 93—94).—A small low-tension arc is struck between pure zinc electrodes, and the flame is spread by means of an oscillating magnetic field placed at right angles. A mixture of equal volumes of nitrogen and oxygen, compressed to 1000 mm. of mercury, is passed through the arc. The zinc from the disintegrated electrodes is burnt completely to extremely finely-divided zinc oxide, which is carried away by the gas stream and

absorbed directly in molten vaseline, through which the gases are eventually bubbled. The whole apparatus must be highly polished to prevent deposition of zinc oxide. Photomicrographs showing the extremely finely-divided and uniform nature of the product compared with zinc oxide treated in a mill are given.

S. COFFEY.

Determination of iodide in mixtures of halides. H. BAINES (J.S.C.I., 1927, 46, 381—382 r).—The following method is shown to be capable of detecting 0.00005 mg. of potassium bromide. To the solution to be tested a few drops of an acid fluorescein solution are added, followed by dilute chlorine water, when the presence of bromide is indicated by a pink coloration. An alternative single-solution method is described which can be made roughly quantitative by colorimetric comparison. The same method can be applied to the detection of iodides, but chlorides, when freed from tenaciously held traces of bromide by recrystallisation in a rapid stream of chlorine, neither respond to the test nor interfere with the detection of bromide.

Bromine analysis. KUBIERSCHKY (Z. angew. Chem., 1927, 40, 1511—1513).—The determination of bromine by partial precipitation with silver nitrate, for which the equation is found to be $d \text{ Br}/\text{Br} = 43 \times d \text{ Cl}/\text{Cl}$, is not accurate when much chlorine is present (e.g., in debrominated liquors or rock salt). Distillation with permanganate or chromate and sulphuric acid and absorbing the bromine with zinc dust is inaccurate, since the bromine is neither completely liberated nor completely absorbed. If manganese dioxide (4 pts.) is added to the permanganate (1 pt.) the bromine is completely liberated and may be completely absorbed in a mixture of sodium hydroxide and hydrogen peroxide. The quantity of absorbed bromine is found by neutralising the alkaline solution with hydrochloric acid, evaporating to dryness, and weighing the mixed sodium bromide and chloride. To determine chlorine in bromine the sample is added to a concentrated solution of a known quantity of sodium bromide, the liberated bromine is boiled off, the solution evaporated to dryness, and the residue weighed. Small quantities (up to 2%) of chlorine in bromides are determined by adding to the latter potassium permanganate and 4*N*-sulphuric acid, boiling off the bromine, filtering, neutralising the filtrate, and determining its chlorine content by titration. It is found that, whilst the bromine is removed in this way, all the chlorine is retained. W. T. K. BRAUNHOLTZ.

Fibrous precipitates. VON WEIMARN.—See V. Compounds of ammonia and carbon dioxide. PAUL and others.—See XIX. Washing of silver bromide. LÜPPO-CRAMER.—See XXI. Ferric salts and sludge activation. MOHLMAN and PALMER. See XXIII.

PATENTS.

Production of synthetic ammonia. K. MÜLLER (B.P. 271,482, 18.5.27. Ger., 19.5.26).—Iron carbide is used as a catalyst at a temperature below that at which it decomposes into iron and carbon, its resistance to heat being increased by the addition of amorphous or colloidal substances (e.g., compounds of the elements of group III of the periodic classification, excepting boron, gallium,

indium, and thallium) or sulphur compounds, and the carbide is converted into a more active form by the addition of 3–5% of cyanogen compounds of the alkali or alkaline-earth metals.

W. G. CAREY.

Purification of gases to be used in the production of ammonia. NORSK HYDRO-ELEKTRISK KVAELSTOF-AKTIESELSKAB (B.P. 266,689, 2.2.27. Nor., 25.2.26).—The mixture of hydrogen and nitrogen is subjected to the action of inorganic compounds dissolved or suspended in liquid anhydrous ammonia, *e.g.*, hygroscopic substances, sodium cyanide, ammonium and calcium nitrates, etc.

H. ROYAL-DAWSON.

Manufacture of ammonium chloride. H. HOWARD, ASSR. to GRASSELLI CHEMICAL Co. (U.S.P. 1,654,125, 27.12.27. Appl., 15.1.25).—Hydrogen chloride and aqueous ammonia are added simultaneously to saturated ammonium chloride solution, the heat of reaction being employed to evaporate the added liquid.

T. S. WHEELER.

Production of anhydrous metal chlorides. I. G. FARBENIND. A.-G. (B.P. 275,945, 23.6.27. Ger., 13.8.26. Addn. to B.P. 259,498; B., 1926, 979).—Metallic oxides or substances containing them (other than magnesia) are mixed with carbonaceous material, *e.g.*, peat, sawdust, etc., and shaped into balls, rods, etc., after which they are dried and coked at below red heat. The porous material so formed, preheated at 100–200°, is then introduced into a furnace and exposed to a current of chlorine.

H. ROYAL-DAWSON.

Preparation of table salt. F. W. HUBER (U.S.P. 1,645,238, 11.10.27. Appl., 20.2.23).—Saturated sodium chloride solution is treated at ordinary temperature with an equal volume of concentrated calcium or magnesium chloride solution, and the precipitated sodium chloride is separated and washed with brine. The moist crystals are then mixed with anhydrous sodium carbonate (2–6%) to form a surface layer of calcium carbonate, after which they can be dried without caking.

T. S. WHEELER.

Purification of alkali cyanide solutions containing sulphur. I. G. FARBENIND. A.-G., ASSEES. OF K. HERRDEGEN (G.P. 444,967, 5.12.25).—Sulphur compounds are precipitated by the addition of bismuth hydroxide or salts.

L. A. COLES.

Manufacture of [partially dehydrated] disodium [hydrogen] phosphate. H. II. MEYERS, ASSR. to ARMOUR FERTILIZER WORKS (U.S.P. 1,648,656, 8.11.27. Appl., 14.1.27).—Concentrated phosphoric acid is mixed with sodium carbonate.

T. S. WHEELER.

Production of alkali hydrides. DEUTSCHE GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER, H. FREUDENBERG, and H. KLOEFFER (B.P. 283,089, 28.9.27. Addn. to B.P. 276,313; B., 1928, 90).—The finely-divided alkali metal is introduced into the reaction space-with or without diluents.

H. ROYAL-DAWSON.

[Simultaneous] production of chromates [and alumina]. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 273,666, 30.6.27).—Sodium chromate and alumina are produced simultaneously by heating at 900–1000°, in an oxidising atmosphere, a mixture of chrome ore and bauxite with sufficient sodium carbonate to form chrom-

ate, aluminate, and ferrate. The product is lixiviated and a solution having d 1.26 (measured hot) is maintained at 100°, or over if in closed vessels, to separate silicic acid as sodium aluminium silicate. The alumina is precipitated by stirring and introducing carbon dioxide and is separated, after which the sodium chromate solution is evaporated to separate the sodium carbonate and is crystallised or converted into dichromate.

W. G. CAREY.

Treatment of leucite and like minerals. P. SPENCE & SONS, LTD., and T. J. I. CRAIG (B.P. 283,087, 29.6.26).—Minerals containing aluminium and potassium oxides are treated with nitric acid, a neutral or basic solution being obtained by removal of free nitric acid and some of the combined acid by heat or by neutralising with alumina or potash, after which iron is eliminated as basic ferric potassium sulphate or by digesting with manganese dioxide, tin oxide, or antimony oxide. The potassium nitrate is separated by crystallising under regulated conditions of temperature and sp. gr., and is washed with a saturated solution of potassium nitrate. Nitric acid is added to the mother-liquor and the aluminium nitrate separated. The mixed nitrates may be cooled below the temperature at which mixed potassium nitrate and aluminium nitrate crystals are formed, and re-warmed to dissolve the aluminium nitrate crystals.

W. G. CAREY.

Recovery of alumina and phosphates from aluminium phosphates. L. MELLERSH-JACKSON. From RHENANIA KUNHEIM VER. CHEM. FABR. A.-G. (B.P. 283,072, 24.6.27).—Aluminium phosphates are mixed with oxides, carbonates, sulphates, or chlorides of alkaline-earth metals and with alkali salts in the proportion of 1 mol. of P_2O_5 to 2 mols. of alkaline-earth metal oxide and 1 mol. of alkali oxide, and 1 mol. of alumina to 1 mol. of alkali oxide, and are heated at 900° or over, the soluble alkali aluminate being extracted with water and the alumina worked up, whilst the phosphate residue is used as a fertiliser. If sulphates or chlorides are used the process is carried out in the presence of steam produced from fuels rich in hydrogen, *e.g.*, water-gas, oil, etc.

W. G. CAREY.

Preparation of an artificial base-exchange substance. E. M. PARTRIDGE and E. W. SCARRITT, ASSRS. to PAIGE & JONES CHEMICAL Co. INC. (U.S.P. 1,656,604, 17.1.28. Appl., 8.12.25).—A wet mixture of sodium silicate and trisodium phosphate is combined with a separately prepared wet mixture of aluminium sulphate and magnesium sulphate to precipitate magnesium phosphate and to form a gel which is dried at a relatively low temperature.

W. G. CAREY.

Manufacture of beryllium and aluminium oxides. C. F. BRUSH, JUN., ASSR. to BRUSH LABORATORIES Co. (U.S.P. 1,656,660, 17.1.28. Appl., 14.12.26).—Beryllium minerals containing aluminium are treated so as to convert the beryllium and aluminium into sulphates, which are freed from impurities and heated, producing a mixture of their oxides.

H. ROYAL-DAWSON.

Production of sulphur dioxide and magnesium compounds from magnesium sulphate. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G., ASSEES. OF F. ROTHE and H. BRENEK (G.P. 445,305, 5.6.25).—Mixtures of

magnesium sulphate with silica or material containing it (1 mol. of SiO_2 per 2 mols. of MgO), or with alumina or ferric oxide, alone or in admixture with one another or with siliceous material, are calcined in the presence of steam, which may be added as such to the flame or formed during combustion by using fuel containing a high proportion of hydrogen. L. A. COLES.

Production of liquid sulphur dioxide from the products of combustion of sulphur or sulphurous pyrites. P. JODECK, Assr. to ALLGEM. GES. F. CHEM. IND. M.B.H. (U.S.P. 1,649,701, 15.11.27. Appl., 12.7.26. Ger., 13.11.24).—The gaseous products are extracted with those hydrocarbons present in naphtha which are soluble in liquid sulphur dioxide and have b.p. 180–300°, and the sulphur dioxide solution obtained is heated to volatilise the gas. T. S. WHEELER.

Fixation of atmospheric nitrogen. J. BRIGHTMORE (B.P. 283,232, 6.7.26).—Preheated air and superheated steam at a pressure of 4–5 atm. are subjected in a closed chamber to a direct or alternating electric current (30 amp. at 80 volts) in the presence of dry, finely-powdered, hydrated lime and calspar, a catalyst of copper or zinc plates also being present. After treatment for about 3 hrs. the materials are transferred to another chamber and subjected alternately to currents of hot and cold air, the hot air being at 480°, and also to electric currents of increased strength. The nitrogenous content may be supplemented by passing an air-blast through an electric-arc flame, then through a jet of water, on to the material. W. G. CAREY.

Activation of carbon and production of carbon disulphide. W. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 283,267, 5.10.26).—Carbon of suitable grain size is treated at a high temperature (800–1000°) with sulphur vapour or with a compound yielding sulphur vapour by decomposition until the required activation is obtained. Carbon disulphide is simultaneously formed. H. ROYAL-DAWSON.

Preparation of nitrates and a dry mixture of chlorine and nitrosyl chloride. W. DOMINIK (U.S.P. 1,658,519, 7.2.28. Appl., 13.3.26. Poland, 11.3.25).—See F.P. 611,652; B., 1927, 330.

Production of alkali-metal xanthates. R. B. CROWELL and G. F. BRECKENRIDGE, Assrs. to AMER. SOLVENTS & CHEMICAL CORP. OF CALIFORNIA (Re-issue 16,866, 31.1.28, of U.S.P. 1,559,504, 27.10.25).—See B., 1926, 13.

Chemical heat storage (U.S.P. 1,656,366).—See I. Highly active carbon blocks (B.P. 266,674).—See II. Ammonium sulphate (B.P. 281,474).—See II. Recovery of ammonia (B.P. 282,927).—See XVI. Aluminium salts (G.P. 444,734).—See XX.

VIII.—GLASS; CERAMICS.

Properties of soda-baryta-silica glasses and a comparison with the corresponding soda-lime- and soda-lead oxide-silica glasses. S. ENGLISH and W. E. S. TURNER (J. Soc. Glass Tech., 1927, 11, 425–432).—In the series of glasses of the molecular type $6\text{SiO}_2 \cdot x\text{BaO} \cdot (2-x)\text{Na}_2\text{O}$ it was difficult to free the glass from seed when the barium-oxide content was as

high as 19.38%, but no difficulty in working occurred until the baryta content was 24.25%. It was possible to incorporate up to 41% of the oxide before a melting temperature of 1400° was exceeded. Annealing temperatures varied practically continuously from 485° for the glass SiO_2 72.86%, BaO 3.03%, Na_2O 23.30%, to 510° for the glass SiO_2 62.67%, BaO 24.25%, Na_2O 11.72%. Linear coefficients of thermal expansion decreased from 1102×10^{-8} for the first member to 884×10^{-8} for the last, whilst density increased continuously from 2.4795 for the first member to 2.8841 for the glass SiO_2 59.97%, BaO 26.15%, Na_2O 11.20%. Curves showing the relationship between the above properties and molecular composition of baryta, lime, and lead oxide glasses are given. A. COUSEN.

Opal glass. Crystal growth and impact brittleness. J. F. HYSLOP (J. Soc. Glass Tech., 1927, 11, 362–369).—In a fluoride opal glass, high viscosity coincides with globular, low viscosity with crystalline, structure of the fluoride particles. When the glass is heated at temperatures below the critical point of Gehlhoff and Thomas (B., 1927, 877), the particle size, for equal times of heating, is inversely proportional to the viscosity. Impact brittleness is chiefly due to the tendency to form sharp angular crystals in the glass, either of silica from the matrix or, with glasses of low viscosity, of the fluoride. One case of brittleness due to the production of heterogeneous glass has been detected. A. COUSEN.

Detection of selenium in decolorised bottle glasses. E. J. C. BOWMAKER and J. D. CAUWOOD (J. Soc. Glass Tech., 1927, 11, 386–392).—The qualitative codeine test for selenium (cf. Schmidt, B., 1914, 748) was developed for use with decolorised bottle glasses. Of substances likely to be present in such glasses only manganese interfered with the test, but even when this element was present the test could be carried out by first precipitating the manganese (after dissolving the glass) by means of bromine. A. COUSEN.

Homogeneity of glass melted in pots on the commercial scale. (Mrs.) G. A. GREEN and W. E. S. TURNER (J. Soc. Glass Tech., 1927, 11, 407–415).—Glasses from a number of pots from different factories, made without mechanical stirring, and including alkali-lead oxide-silica, alkali-lime-silica, and potash-baryta-lime-boric oxide-silica glasses were proved to be very homogeneous by an analysis of samples from the top, middle, and bottom of the pot in each case. A. COUSEN.

Manufacture and use of glasshouse pots in Great Britain. W. E. S. TURNER (J. Soc. Glass Tech., 1927, 11, 415–424).—The tabulated results of a survey of the methods of pot manufacture of 14 firms are given, the details of which include particulars of size and method of making, preparation of clay, and the drying, arching, and record of the life of the pots. A. COUSEN.

Stourbridge fireclays and the manufacture of glasshouse pots. M. H. EDWARDS (J. Soc. Glass Tech., 1927, 11, 400–405).—An account of the mining and subsequent treatment of Stourbridge clay employed for the manufacture of glasshouse pots. A. COUSEN.

Measurement of plasticity [of clays]. T. W. TALWALKAR and C. W. PARMELEE (J. Amer. Ceram. Soc., 1927, 10, 670—685).—Methods of measuring plasticity are examined, and objection is raised to the significance given by Bingham and Green (B., 1919, 495 A) to the term plasticity, a more limited conception of this quality of clays being preferred. The stress-strain relations in shear of carefully prepared specimens of clay have been determined by means of a torsion machine. It is shown that for the clays examined the relation of stress to strain in shear is the same as that in compression, and that between certain limits the strain is proportional to the stress; beyond this limiting value, which is not the same as the limit of elasticity, the rate of deformation becomes comparatively great. Addition of water to any clay or of graphite to a non-plastic clay increases the rate of deformation beyond the proportional limit. It is suggested that the plasticity index for any clay-water mixture may be expressed as the ratio of total deformation at the point of failure to the average stress beyond the proportional limit. A. T. GREEN.

Quantitative microscopic analysis of commercial felspar. H. INSLEY (J. Amer. Ceram. Soc., 1927, 10, 651—669).—A method for the rapid determination of the constituents of ground commercial felspar is based on the separation of the mineral constituents according to their indices of refraction and the determination of their volume percentages. This method, which gives close agreement with the data calculated from chemical analyses, involves the use of a liquid of index of refraction 1.540 for distinguishing quartz from felspar, and a liquid of index 1.528 for distinguishing potash felspar from plagioclase. Another method in which a partially fused sample is examined microscopically for the quartz content is described. The results of microscopical analysis of nineteen samples of felspar are tabulated. Although chemical analysis is the more accurate, the microscopical methods are valuable in works control, when rapid determinations are necessary.

A. T. GREEN.

Refractory material for vertical retorts. STEINHOFF (Gas World, 1928, 88, 72—73).—See B., 1927, 937.

PATENTS.

Continuous kilns for burning [ceramic products] and like purposes. A. E. OSMAN (B.P. 283,009, 19.2.27).—The kiln chambers have upcast shafts at each end, through which the combustion gases from furnaces on either side thereof pass into the upper part of each chamber. The gases from a preceding chamber also pass into one of the shafts, mixing with the combustion gases therein. The hot gases pass down through the chamber into flues running below the centre of the floor, or through perforations in the floor at each side into flues running along each side of the chamber. A suitable arrangement of flues permits the passage of gases or heated air from a chamber being cooled down to another being heated up. A. B. MANNING.

Extraction of clay and other argillaceous raw materials with acids. I. G. FARBENIND. A.-G. (B.P. 278,370, 29.9.27. Ger., 29.9.26).—The acid used for the extraction percolates through a large quantity (100 tons) of the raw material to be treated, and main-

tains a certain liquid level above the charge, the passage of acid used being so regulated by its inflow and outflow that the heat of reaction causes a temperature of at least 105° to be attained in the downwardly travelling zone of maximum reaction in the charge.

H. ROYAL-DAWSON.

Cementing of porcelain. Cementing of porcelain and metal. Cementing of porcelain, and porcelain and iron. STUDIEN-GES. F. WIRTS. U. IND. M.B.H. (B.P. 260,592, and Addn. B.P. 260,593 and 274,408, [A—C] 26.10.26. Ger., [A, B] 27.10.25, [C] 17.7.26).—In (A) a rubber mass containing vulcanising accelerators is used. The whole of the rubber is combined with sulphur and contains filling or other materials to increase its tensile strength. (B) A similar cement is used, but the metal parts are first coated with hard rubber. (C) Sulphur is replaced by selenium or its compounds. A suitable cement for all the purposes enumerated consists of 90% of pure Para rubber, 2% of litharge, 2% of selenium oxide, 3—4% of vulcanising accelerators, e.g., piperidine, and 2—3% of zinc oxide or finely-ground soot. W. G. CAREY.

Manufacture of refractories. M. J. RENTSCHLER, Assr. to W. R. and A. N. JEAVONS (U.S.P. 1,653,123, 20.12.27. Appl., 13.12.20).—A mixture of clay, carbon, firebrick, and water is formed, dried, and coated with a composition containing fireclay, calcium carbonate, sand, sodium silicate, felspar, and water, so that on firing a glaze is formed which protects the carbon body in an oxidising atmosphere. T. S. WHEELER.

Production of a carbon-bonded refractory. E. L. HAUMAN, Assr. to ELECTRO REFRACTORIES CORP. (U.S.P. 1,653,850, 27.12.27. Appl., 28.4.26).—A crucible is formed of graphite, silicon carbide, a salt, and coal tar, and is lined with a mixture of bonding clay, magnesite, and coal tar. The outer portion is then impregnated with borax solution so that when the crucible is heated at 900° in an oxidising atmosphere the carbon is eliminated only from the lining, which is thus rendered resistant to the action of molten steel.

T. S. WHEELER.

Manufacture of an abrasive article. D. F. WEBSTER, Assr. to NORTON Co. (U.S.P. 1,655,396, 3.1.28. Appl., 14.4.26).—An intimate mixture is made of the abrasive grains, rubber, and sulphur, together with not more than 30% of a synthetic resin, e.g., bakelite. The mixture is then shaped and vulcanised. F. R. ENNOS.

Production of [cellular] heat-insulating material. A. U. WESTFELT (B.P. 283,055, 28.5.27).—In the production of firebricks, water-glass is mixed with potassium chlorate and, if desired, a powdered silicate, and heated.

H. ROYAL-DAWSON.

IX.—BUILDING MATERIALS.

Retarding the setting time of cement by the addition of small quantities of lead monoxide. B. GARRE (Zement, 1927, 16, 469; Chem. Zentr., 1927, II, 726).—The setting time is retarded considerably by the addition of 0.001% of lead monoxide, further additions retarding it still more until with about 1% hardening is incomplete. L. A. COLES.

Suggested standard method of slaking in testing building limes. A. D. COWPER and J. F. WILLIAMS (J.S.C.I., 1928, 47, 31—35 r).—A standard isothermal method for slaking building limes for the purposes of formal tests is proposed, using a large excess of water and rapid stirring, the quicklime being crushed to $\frac{1}{2}$ in. size. Tests at 50° and at 100° are made, and the product of higher volume-yield is chosen for further tests; or a temperature designated by the manufacturer may be used. Results are given for a number of British limes, with analyses, plasticity figures, and comparative results for dry and uncontrolled slaking.

Wood preservation. VI. L. P. CURTIN and W. THORDARSON (Ind. Eng. Chem., 1928, 20, 28—30); cf. B., 1927, 750, 909, 938; 1928, 93).—Further species of wood-rotting fungi have been grown in nutrient media containing sodium alizarinsulphouate and methyl-orange. In all cases sufficient acid was formed to affect the former indicator, and in some cases, notably with *Merulius lachrymans*, a fungus which causes large losses of timber by "dry rot," the culture became acid to methyl-orange. Tables are given showing the "killing points" (minimum concentration that completely inhibits the growth of a test fungus, viz., *Fomes annosus*) of a number of inorganic preservatives, also the toxicity to various fungi of powdered zinc meta-arsenite ("killing point" 0.10) at concentrations up to 0.3%. From the practical point of view zinc meta-arsenite is the most promising of the preservatives studied; it does not increase the electrical conductivity of wood, has no corrosive action on iron, and may be used in standard pressure-treating equipment. Moreover, field tests have shown that wood containing zinc meta-arsenite suffers little loss of preservative during prolonged exposure to the weather.

D. J. NORMAN.

PATENTS.

Manufacture of Portland cement in a rotary kiln. G. MARTIN (B.P. 282,868, 27.9.26).—The output of a rotary kiln is increased by varying the rate of travel of the gases through the zones into which a kiln is naturally divided, the speeds being in some zones substantially greater, in some less, and in some the same as those prevailing in practice. Thus the gases are caused to traverse internal baffles, so that the average linear speed is greater than 22 ft./sec. in the drying zone, and greater than 30 ft./sec. in the clinkering and decarbonating zones.

W. G. CAREY.

Calcining of cement or lime. E. MEIER (G.P. 445,554, 7.9.24).—The finely-divided material falls by gravity down a shaft kiln which is bellied at the combustion zone and at a zone below it from which the waste gases are withdrawn. The combustion gases enter the kiln, and the waste gases leave it in a direction at right angles to that of the falling particles, the waste gases being free from dust.

L. A. COLES.

Grinding of cement and similar materials. C. PONTOPIDAN (B.P. 283,091, 8.10.27).—In order to absorb the heat produced by grinding and to maintain the temperature below that at which the cement becomes inclined to adhere to the grinding surfaces, a liquid which will evaporate and so abstract heat is added during the final stages through the discharge end of the grinding

machine by means of a pipe directed axially and terminating in an atomiser. The liquid may be supplied through holes in the casing by scoop or blade wheels, and material such as calcium chloride, calcium sulphate, or tannin may be suspended or dissolved in the liquid.

W. G. CAREY.

Preparation of emulsions or suspensions [for roads etc.]. G. BAUME, P. CHAMBIGE, and D. BOUTIER (B.P. 255,074, 7.7.26. Fr., 9.7.25).—The emulsion is prepared by adding to the basic mixture consisting of bitumens, tars, pitch, tar oil, etc., a stabiliser, e.g., stearine pitch, vegetable or mineral oils, a colloid protector, e.g., gelatin, glues, gums, casein, or soaps, and to impart elastic properties tartrates, esters of glycerin or aromatic alcohols. The emulsifying agent is added with a small proportion of water to the basic mixture, and finally the rest of the water is added. These emulsions have a narrow critical temperature range of 5—10°.

A. C. MONKHOUSE.

[Press table for] the manufacture of bricks. SCHEIDHAUER & GIESSENG A.-G., and H. ACKERMANN (B.P. 282,711, 23.9.27. Ger., 31.12.26. Addn. to B.P. 267,948).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Behaviour of steel under prolonged stress at high temperatures. A. POMP (Chem. Fabr., 1928, 53—54).—Recent work on the "creeping" of steel at high temperatures is reviewed and the necessity of determining the "permanent strength" of steels which are to be used for high-pressure work at elevated temperatures is emphasised. The "permanent strength" of a steel is defined as the highest load which the steel will support at any given temperature without increasing in length after 3—6 hrs. by more than 0.001%/hr. For plain carbon steels this value remains fairly constant up to 300°, then falls very rapidly with rise in temperature; the permanent strength and the rate at which it decreases with rise of temperature increase with the carbon content of the steel up to the eutectoid point, provided that the pearlite constituent is in the lamellar form. Steels in which the pearlite is in a granular form have a much lower permanent strength.

A. R. POWELL.

Hardening by compression and the brittleness of steel. Existence of a limit of brittleness. P. DE-JEAN (Rev. Mét., 1927, 24, 415—417).—When steel is subjected to compression, so long as the load does not exceed a certain critical value, the resilience remains constant irrespective of any previous compression below the critical value to which the metal has been subjected. When the load exceeds this value the resilience falls sharply and the metal exhibits a slight quiver indicating that it has passed from the resilient to the brittle state. The load required to produce the brittle state in a mild steel is greatest after annealing at 900°, quenching in cold water, annealing at 700°, and cooling slowly; forging reduces the critical load, and prolonged annealing at 950° followed by slow cooling in air reduces it to about half that of the metal annealed at 900°.

A. R. POWELL.

Cast iron resistant to acids and alkalis. H. G. HAASE (*Stahl u. Eisen*, 1927, 47, 2112—2117).—The most satisfactory resistance to attack by mineral acids is shown by cast iron of the following composition: 3.3—3.5% C, 1.3—1.4% Si, about 0.75% Mn, 0.3—0.5% Ni, not more than 0.2—0.3% P, and not more than 0.07% S. Increase of sulphur is particularly deleterious, more manganese increases the rate of corrosion relatively slightly, with silicon maxima occur in the corrosion curve at 2.5 and 7% Si, and with phosphorus and nickel little difference is noticeable in the rate of corrosion with double the above amounts. The cast iron most resistant to alkali contains 3.3—3.5% C, 1.2—1.4% Si, 0.3—0.4% Mn, not more than 0.3% P, not more than 0.08% S, and 0.3—0.5% Ni. Increase of phosphorus reduces the resistance to attack by alkali hydroxides, as also does increase of silicon to a maximum of 7% Si. Metal with the casting skin intact is more resistant than metal from which this skin has been removed.

A. R. POWELL.

[Determination of] phosphorus and silicon in ferrophosphorus. L. E. PITZER (*Chemist-Analyst*, 1926, No. 47, 8).—The sample (0.25 g.) is heated with 60% perchloric acid (35 c.c.), cooled, made slightly ammoniacal, treated with a slight excess of concentrated nitric acid, and diluted to 75 c.c. The phosphorus is determined with molybdate. For the determination of silicon, treatment with perchloric acid is advised.

CHEMICAL ABSTRACTS.

Analysis of chrome iron ore. E. DITTLER (*Z. angew. Chem.*, 1928, 41, 132—133).—Fusion of this ore with sodium carbonate is never complete, and the analysis is best carried out in the following manner. The finely-ground mineral is fused with ten times its weight of sodium peroxide in a silver crucible, the melt dissolved in water, acidified with hydrochloric acid, and the precipitated silver and silver chloride removed. The filtrate, to which a few c.c. of hydrogen peroxide have been added to reduce the chromium present, is evaporated to dryness and the silicic acid removed by evaporation with hydrofluoric acid; the residue is decomposed with sodium carbonate and a little potassium nitrate, and, after treatment with hydrogen peroxide, the combined oxides of aluminium, iron, and chromium are precipitated with ammonia solution. Provided that excess of hydrogen peroxide is removed before precipitation, all the chromium is obtained as oxide. The three oxides are treated with sodium peroxide, when the iron remains as insoluble residue and the chromate is determined volumetrically in the filtrate after conversion of the manganese present into manganese dioxide, whilst the aluminium content is obtained by difference. In the filtrate from the three oxides, calcium, magnesium, and manganese are determined by the ordinary methods.

W. J. POWELL.

Concentration of ore from the Read-Rosebery mines of Electrolytic Zinc Co. of Australasia, Ltd. E. H. FRASER (*Proc. Austral. Inst. Min. Met.*, 1927, [66], 179—186).—The ore, consisting of blende, galena, and pyrites in a schistose gangue, is ground in a 0.5% solution of sodium carbonate in rolls and Hardinge or tube mills so that 90% passes 200-mesh. The mill discharge

passes to classifiers and the overflow to thickeners, thence to the lead flotation plant. The pulp ratio in this plant is kept at 1:1, the solution containing 0.35% Na_2CO_3 ; the lead sulphide is floated with 0.3 lb./ton of coal-tar distillate in a series of nine sub-aeration cells, and the concentrate cleaned in a series of eight similar cells. The final dried product contains 46% Pb, 19% Zn, 48.5 oz./ton of silver, and 6 dwt./ton of gold. The tailings from the lead plant pass to the zinc plant, the pulp density having been reduced to 1:3 and the alkalinity to 0.05—0.1% Na_2CO_3 ; zinc sulphide is floated with 0.75 lb./ton of coal-tar distillate and 1.5 lb./ton of copper sulphate, the pulp being maintained at 50° by steam injection. The zinc concentrate contains 53% Zn, 6% Pb, 7.4 oz./ton of silver, and 3 dwt./ton of gold. The recovery of zinc is 86% and of lead 60%, whilst 92% of the silver and 70% of the gold are recovered in both concentrates; the total soda consumption is 11 lb./ton of ore.

A. R. POWELL.

Drying and roasting of zinc [sulphide] concentrate at Zeehan Works of Electrolytic Zinc Co. of Australasia, Ltd. E. H. FRASER (*Proc. Austral. Inst. Min. Met.*, 1927, [66], 173—177).—The fine flotation concentrate obtained as described in the preceding abstract is fed to a dryer 45 ft. long and 14 ft. wide, fired with wood fuel and mechanically rabbled. The discharge from the furnace contains 2—3% of moisture, and is fed directly by means of conveyors and elevators to four 7-hearth Herreshoff furnaces, each hearth of which is rabbled mechanically with a cast chromium-steel (27% Cr) rabble provided with eight teeth and rotated 52 times/hr. The furnace is fed with preheated air in such a way that the third hearth is kept at 850°, whilst the seventh is at 600°, no fuel being used. The discharge from the furnace, after cooling somewhat, is damped down and sent to the dissolving plant for the production of electrolytic zinc; it contains 58% Zn, 5.4% Pb, 2.6% S as sulphate, and 4.8% S as sulphides. About 20% of the lead input to the furnace is recovered in fume, and the flue gases contain 2—2.5% SO_2 .

A. R. POWELL.

Theory of the age-hardening process in constructal and montegal [aluminium alloys]. W. GUERTLER (*Z. Metallk.*, 1927, 19, 488—491).—The constituent in constructal, which renders the alloy amenable to age-hardening, is the compound MgZn_2 , which is not decomposed by aluminium but forms with it a eutectic, m.p. 480°, and a limited series of solid solutions containing up to 28% MgZn_2 at 480° and 4—5% at 20°. For practical purposes the alloy should contain 5—12% MgZn_2 , and the magnesium and zinc content should be so arranged that neither is in excess of the amount required to form this compound. In order to obtain a highly-dispersed separation of the compound during ageing, a temperature above the ordinary is required, the exact temperature depending on the amount of MgZn_2 present. Correct adjustment of composition and heat treatment gives an alloy having a higher tensile strength and ductility than duralumin. Montegal contains calcium silicide as the hardening constituent, and after heat treatment has a tensile strength almost equal to that of duralumin but is

much more ductile, can be rolled into very fine foil, and has a high bending strength and Brinchen value.

A. R. POWELL.

Tensile strength and electrical conductivity of recrystallised aluminium wire, and its practical application in rolling the metal. H. BOHNER (*Z. Metallk.*, 1928, 20, 8—13).—The tensile strength of aluminium wire falls rapidly and the electrical conductivity increases appreciably after annealing for 1 min. at 300° or above. Between 300° and 400° the tensile strength decreases slowly and the conductivity increases slightly over a period of 5 min.: at higher temperatures there is practically no further change after the first minute. With aluminium containing 98—99% Al prolonged annealing at 575° has little effect on the strength or conductivity, whereas with 99.4—99.7% Al the tensile strength decreases with time of annealing at 575° owing to the development of a very coarsely crystalline structure. When soft 98—98.5% Al is annealed at 500° the tensile strength and conductivity remain constant for 45 min., after which the former suddenly increases slightly and the latter decreases. In all the specimens tested the tensile strength increased with the temperature of recrystallisation provided that coarse crystals did not develop; the electrical conductivity showed a corresponding decrease. Under the same conditions of annealing the tensile strength is lower and the conductivity higher the higher the purity of the metal. For the preparation of aluminium wire for electrical conductors the most satisfactory annealing temperature is 350°. The optimum conditions for casting and rolling the metal are briefly outlined.

A. R. POWELL.

Experimental determination of the curve of critical dispersion of the alloy "lantal." K. L. MEISSNER (*Z. Metallk.*, 1928, 20, 16—18).—Specimens of lantal (4% Cu, 2% Si, and 94% Al) were annealed at 510—515° for 20—30 min., quenched in water, and aged for prolonged periods at temperatures between 125° and 220°, the hardness and bending strength being determined periodically during the ageing. At 125—160° the hardness increases from 90 to a more or less constant value of 130 in 12—1 days according to the temperature, at 175° maximum hardness is reached in 12 hrs., and further ageing results in a steady softening of the metal; at 200° the maximum hardness is only 110 and is reached in 5 hrs., and at 220° it is 100, reached in 3 hrs. The bending strength falls slowly to a minimum in 12 hrs. at 175°, then increases again almost to its original value after 24 hrs. The curve of critical dispersion is obtained by plotting the ageing temperature against the time required to reach maximum hardness; it is a hyperbolic curve which falls steeply between 220° and 180°, and very slowly between 150° and 130°. The results obtained in the investigation indicate that the most satisfactory ageing temperature for lantal is 160°, 30 hrs. being required for maximum hardness. Rapid coagulation of the copper constituent occurs above the critical ageing temperature, but at lower temperatures prolonged annealing has little deleterious effect on the properties of the metal.

A. R. POWELL.

Aluminium bronze. J. BOULDOIRES (*Rev. Mét.*,

1927, 24, 357—376, 463—473).—Sand-cast aluminium bronzes containing 80—95% Cu were annealed at 850° for 5 hrs., cooled to 20° during 15 hrs., and heating and cooling curves obtained for the following properties: electrical resistance, thermo-electric power, density, hardness, and expansion. In addition, the alloys were examined by thermal and micrographic analysis. In the case of alloys consisting entirely of α -solid solution, an anomaly was observed in the heating curves at 290—310°, but not in the cooling curves. Alloys consisting of α + eutectoid showed a transformation at 510—520° both on heating and cooling, and those consisting of γ + eutectoid one at 515° on heating and at 493° on cooling; both types of alloys in addition showed the eutectoid transformations at 565—570° and at 690—780°. The change of direction of the various curves for the low-temperature transformations is more marked the higher the aluminium content of the alloys and the more slowly the metal has been cooled after annealing. After rapid cooling or quenching many of these transformations are suppressed, and no deviations occur in subsequent heating and cooling curves. The thermal effect at 300° is not accompanied by any change in the structure or physical properties of the alloy, but that at 520° is accompanied by changes in the density, electrical resistance, and other physical properties, although no structural change can be detected. Quenching from above the eutectoid point (560°) decreases the resistance and hardness of most aluminium bronzes.

A. R. POWELL.

Determination of lead in manganese bronze. F. B. DIANA (*Chemist-Analyst*, 1927, No. 47, 6—7).—The alloy is treated with ferric nitrate solution, then with nitric acid, and, after boiling to remove excess of acid and diluting, the lead is determined electrolytically as dioxide.

CHEMICAL ABSTRACTS.

Analysis of lead-base bearing metal. B. PAXTON (*Chemist-Analyst*, 1926, No. 47, 3).—The sample (1 g.) is decomposed with sulphuric acid (15 c.c.) and potassium sulphate (2 g.), and the cool liquid is diluted with water (100 c.c.) and boiled for 12 min. with hydrochloric acid (10 c.c., concentrated). After rapid cooling, water (200 c.c.) is added, and the antimony titrated with 0.1N-permanganate solution. Lead sulphate is collected from the cold titrated solution, washed, dissolved in ammonium acetate solution, and titrated with molybdate. To the filtrate from the lead sulphate are added concentrated hydrochloric acid (25 c.c.), iron nails, and sufficient of a solution (made by dissolving 1 g. of antimony in 20 c.c. of concentrated sulphuric acid, diluting to 100 c.c. with dilute hydrochloric acid, and adding enough hydrochloric acid to dissolve oxychloride) to dissolve up to 0.15 g. of antimony that may have been precipitated by the iron. The mixture is boiled for 25 min., cooled in carbon dioxide, filtered, and the tin titrated with 0.1N-iodine solution.

CHEMICAL ABSTRACTS.

Intercrystalline brittleness of lead. O. HAEHNEL (*Z. Metallk.*, 1927, 19, 492—496).—Lead cable and water-pipes become rapidly brittle when subjected to vibration owing to loosening of the intercrystalline bond. The rate at which this occurs with pure lead increases with

the rate of vibration and with the temperature. Exposure to temperatures between 40° and 100° results in a slow development of brittleness due to crystal growth; higher temperatures rapidly cause a breakdown of the adhesion between the crystals. Compression, tension, or torsion alone does not cause brittleness, but in conjunction with vibration stresses they increase the rate of breakdown. The resistance to intercrystalline fracture is increased 7–10 times by the addition of 3% Sn or 1% Sb to the lead. A. R. POWELL.

Analysis of acid-resisting bronze, antimonial lead, and babbitt metal. I. R. F. SMITH (Pulp and Paper Mag., 1928, 26, 105–106).—The bronze (1 g.) is dissolved in 30 c.c. of 1:1 nitric acid, the solution evaporated nearly to dryness, diluted to 100 c.c. with hot water, and filtered, and the precipitate of metastannic acid is ignited and weighed. The filtrate is neutralised with ammonia solution, treated with 3 c.c. of nitric acid, diluted to 200 c.c., and electrolysed for 1½–2 hrs. with 4–6 volts and 3–5 amp. using a rotating, cylindrical, platinum gauze cathode inside a similar but larger anode. Copper is deposited on the cathode and lead peroxide on the anode. Iron, manganese, and aluminium are removed from the spent electrolyte by addition of ammonia solution, ammonium chloride, and bromine, and the zinc in the filtrate is precipitated as zinc ammonium phosphate and subsequently weighed as the pyrophosphate. An alternative method involving precipitation of the lead as sulphate and of the copper as sulphide by means of thiosulphate, followed by volumetric determination by the iodide method, is described. A. R. POWELL.

Detection of baser admixtures in gold plates. G. TAMMANN and O. LOEBICH (Z. anorg. Chem., 1927, 168, 255–263).—The presence of 0.1% of iron, tin, or antimony in a gold plate can be detected by observing the colour of the surface film produced by heating the plate at 600°. At this concentration the detection of aluminium and tellurium is, however, somewhat uncertain, but by observation of the poisonous action of the plate on a culture of *Bacterium gossypii* or *B. Brassicae* in contact with it, 0.1% of antimony or tellurium will be revealed. If, however, the impurity forms mixed crystals with gold, the bacterial method is, in general, useless. Iron, tin, and antimony cause the appearance of spots when the gold is subjected to the action of dry iodine vapour, but spots are also frequently due to foreign bodies embedded in the surface in such processes as rolling and polishing. The precipitation of radium fluoride from acid solution by a gold sheet is not very satisfactory as a means of detecting foreign metals, as gold itself adsorbs the fluoride to a slight extent, and, further, many metals oxidise before precipitation can occur. R. CUTHILL.

PATENTS.

Heating of smelting furnaces. C. BRACKELSBURG (B.P. 283,381, 13.4.27. Ger., 9.3.27).—Combustion of pulverised fuel is caused to take place directly in a rotary smelting furnace, without a preliminary combustion chamber. A relatively high pressure (400 mm.) of air is used to effect rapid combustion and to carry ash through and out of the furnace, the air at high

pressure being preferably supplied as an envelope to the air-fuel mixture as it leaves the burner nozzle at normal pressure. C. A. KING.

Apparatus for the concentration of ores and other minerals and materials. J. A. CHAMBERS (U.S.P. 1,656,042, 10.1.28. Appl., 9.11.25. S. Africa, 9.9.25).—A pan for wet concentration is provided with means for introducing the ores, an outlet for the lighter constituents and the liquid, and means for withdrawing the concentrates from its lower end. The pan is carried by a plate supported by balls upon a base plate, and is moved in a circular path by mechanism engaging its centre directly. Rotation of the pan about its own axis is prevented by an arm pivoted at a distance and having a sliding connexion with one side of the pan. H. HOLMES.

Ore-grinding machine. C. E. PAYNE (U.S.P. 1,656,756, 17.1.28. Appl., 5.5.27).—A rotary cone is mounted within a stepped casing forming a series of superposed grinding chambers of increasing diameter from top to bottom. The cone carries annular flanges, one projecting into each chamber, and wear blocks are fixed to radial ribs projecting upwards from the flanges. H. HOLMES.

Apparatus for the recovery of minerals from ore. W. C. TRACY (U.S.P. 1,657,097, 24.1.28. Appl., 3.8.25).—On the bottom of a tank is mounted a flat horizontal coil provided with a series of oppositely diverging perforations, and around the inner periphery of the tank is a perforated helical coil for emitting air currents transversely across the tank. Means are provided for supplying air under pressure to the coils. M. E. NOTTAGE.

Smelting of iron in foundry shaft furnaces. F. W. CORSALLI (B.P. 254,727, 30.6.26. Ger., 30.6.25).—For the production of cast iron containing less than 3% C, the air-blast is increased beyond the pressure necessary to produce grey iron, and refining materials, e.g., silicon, are protected in a sheath until the smelting zone is reached. It is preferred to charge only metal into the main shaft, and fuel into subsidiary side shafts communicating at the bottom with the main shaft. C. A. KING.

Manufacture of malleable iron castings. H. A. SCHWARTZ, ASSR. to NAT. MALLEABLE & STEEL CASTINGS Co. (U.S.P. 1,656,535, 17.1.28. Appl., 23.3.26).—Molten iron is introduced into an acid-lined electric furnace, and, after casting, the metal is subjected to a shortened annealing process. A desulphurising agent is added at a stage prior to casting to nullify the effects of sulphur. H. ROYAL-DAWSON.

Manufacture of iron and steel or iron alloys and steel alloys. F. KRUPP A.-G. (B.P. 268,716, 2.9.26. Ger., 3.4.26).—During manufacture, iron or one of its alloys is deoxidised by means of suitable reagents to an oxygen content of less than 0.005%, which is determined by the absence of any substantial decrease of "tenacity of the notched bar" on subsequent ageing of the metal. Up to 0.5% of nickel, cobalt, chromium, tungsten, vanadium, or molybdenum may be added. C. A. KING.

Pickling of chromium-iron alloys. L. SCHULTE, Assr. to ALLEGHENY STEEL Co. (U.S.P. 1,657,228, 24.1.28. Appl., 2.6.26).—The scale is activated in a solution containing hydrofluoric acid and the salt of a metal positive to iron and chromium, and is then dissolved in a solution containing nitric acid.

M. E. NOTTAGE.

Corrosion-resistant [copper-manganese] alloys. M. G. CORSON, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,657,957, 31.1.28. Appl., 13.4.26).—Alloys resistant to attack by inorganic acids are made by alloying copper with 35–55% of manganese.

F. G. CROSSE.

Flux applied in soldering aluminium and its alloys. O. SPENGLER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,653,482, 20.12.27. Appl., 24.12.23. Ger., 23.6.22).—A mixture of sodium fluoride with lithium, calcium, and zinc chlorides is claimed. T. S. WHEELER.

Manufacture of [dumet] bimetallic wire. B. F. STEEL, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,653,378, 20.12.27. Appl., 1.7.22).—The process of manufacture of the wire is modified in that it is heated rapidly during the first annealing to expand any gas trapped between the core and sheath and thus render visible a defect of this kind.

T. S. WHEELER.

Sintered hard-metal alloys and articles made thereof. F. KRUPP A.-G. (B.P. 279,376, 18.3.27. Ger., 25.10.26).—Material for the production of tools etc. consists of a mixture of the carbides of a hard metal, *e.g.*, tungsten, and of at least one softer metal, *e.g.*, iron, the grains being of such size (less than 1×10^{-4} mm.) that the fracture of the tool reveals no pores under a magnification of 50 diameters. Such comminuted raw material is produced by separately grinding tungsten and iron in a ball mill, carburising the tungsten, and regrinding for at least 50 hrs., afterwards mixing, pressing, and sintering the components. C. A. KING.

Treatment of titanium ores. I. G. FARBENIND. A.-G. (B.P. 267,547, 11.3.27. Ger., 11.3.26).—Finely-ground ilmenite is heated at 180° under pressure with 20–40% sulphuric acid containing sufficient titanous sulphate to reduce the ferric iron in the ilmenite to the ferrous condition. Titanium dioxide is obtained from the resulting solution by hydrolysis without further dilution. The titanous sulphate solution is made by reducing a strongly acid solution containing quadrivalent titanium with the product obtained by heating ilmenite with coal or reducing gases at high temperatures.

A. R. POWELL.

Alloy having a high electrical resistance. Y. KAMISHIMA (B.P. 283,354, 9.2.27).—A non-oxidisable alloy with good magnetic properties contains 1–5% Ni, 7–20% Cr, and 1–6% Si, the remainder being iron and impurities, but free from manganese and having a very low carbon content.

F. G. CROSSE.

Alloy. E. KIAM, Assr. to M. G. COLLINS (U.S.P. 1,653,630, 27.12.27. Appl., 25.2.22. Renewed 19.5.27).—The alloy consists of 30–65% Ni, 15–40% W, 15–30% Cr, 1–8% Mn, and 5–0.5% Mg.

F. G. CROSSE.

Composition of matter [alloy]. T. DE LA CONCHA, Assr. to P. A. HUGHES (U.S.P. 1,653,088, 20.12.27.

Appl., 27.10.26).—The alloy is composed of 30% Pb, 50% Sn, 12% Zn, 5% Al, and 3% of German silver.

F. G. CROSSE.

Production of rare metals and their alloys. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,648,954, 15.11.27. Appl., 29.9.21).—An alloy of a rare metal, *e.g.*, thorium, uranium, or cerium, with aluminium is more stable in air than the pure metal, and can readily be prepared by the thermit process or by treating with aluminium a solution of the double chloride of the rare metal and ammonium, *e.g.*, ammonium thorium chloride, in fused potassium and sodium chlorides. The metal is recovered from the alloy by volatilisation of the aluminium.

T. S. WHEELER.

Production by electrolysis of thin superposed nickel sheets and separation of them one from another. E. BREUNING and O. SCHNEIDER (B.P. 275,221, 22.7.27. Ger., 27.7.26).—A layer of nickel is deposited electrolytically and its surface polarised (*e.g.*, by immersion of the rinsed film in concentrated nitric acid or in a solution of chromic acid, or electrically by means of a 5% sodium hydroxide bath), the operations being repeated for each superposed layer; the layers are then separated by means of weak acid.

H. ROYAL-DAWSON.

Electrodeposition of metals [plating on aluminium and its alloys]. H. WEBB and S. O'BRIEN & PARTNERS, LTD. (B.P. 283,288, 9.10.26).—The metal (aluminium) is cleaned by immersion in sodium hydroxide solution, pickled in concentrated nitric acid, and plated with copper from a copper nitrate bath; alternatively it may be pickled in 5–10% sulphuric acid and plated directly with copper, nickel, or zinc from a slightly acid sulphate solution. In either case the plated article is dried in warm sawdust and embedded in silver sand heated at 180° for a period which varies with the thickness of the metal. After heat-treatment the articles may be plated again with the same or a different metal, *e.g.*, copper-plated aluminium may be coated with silver or gold by plating in the usual cyanide baths.

A. R. POWELL.

Treatment of ores. D. C. HARE, Assr. to INTERNAT. REDUCTION Co. (U.S.P. 1,657,622, 31.1.28. Appl., 26.5.24).—See B.P. 234,524; B., 1925, 676.

Apparatus for treatment of steel ingots. J. V. ELLIS (U.S.P. 1,657,350, 24.1.28. Appl., 12.4.26. U.K., 18.3.26).—See B.P. 268,123; B., 1928, 416.

High-percentage magnesium alloys. I. G. FARBENIND. A.-G., Assees. of CHEM. FABR. GRIESHEIM-ELEKTRON (B.P. 254,741, 2.7.26. Ger., 3.7.25).—See F.P. 618,425; B., 1927, 881.

[Aluminium-silicon] alloys. A. G. C. GWYER and H. W. L. PHILLIPS, Assr. to BRIT. ALUMINIUM Co., LTD. (U.S.P. 1,657,389, 24.1.28. Appl., 22.1.24. U.K., 22.1.23).—See B.P. 219,346; B., 1924, 793.

Magnetic alloy and its application in the manufacture of telegraphic and telephonic cables. W. S. SMITH and H. J. GARNETT (U.S.P. 1,656,959, 24.1.28. Appl., 8.1.26. U.K., 20.11.25).—See B.P. 266,066; B., 1927, 303.

Cementing of porcelain and metal (B.P. 260,592—3 and 274,408).—See VIII.

Rubber coatings on metal (B.P. 283,049).—See XIV.

XI.—ELECTROTECHNICS.

Zinc oxide for ointments. DÖRZBACH.—See VII.

Gelatin. OKA.—See XV.

PATENTS.

[Recording] photometric apparatus. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of G. A. LONG (B.P. 275,597, 22.7.27. U.S., 9.8.26).—A recording photometer comprises a reflector which can be rotated about a lighting unit so as to reflect light upon a photoelectric cell, an oscillograph and a synchronised photographic recorder rotating across the beam of light reflected by the oscillograph mirror. J. S. G. THOMAS.

Electric heating of travelling material. AJAX ELECTROTHERMIC CORP., Assees. of E. F. NORTHRUP (B.P. 279,779, 20.4.27. U.S., 28.10.26).—Alternating current is passed through the travelling material from parallel-connected contacts, substantially equal inductances being arranged in series with the individual contacts in order to distribute the current uniformly. J. S. G. THOMAS.

Electric accumulators. G. FROMONT (B.P. 268,828, 2.4.27. Fr., 2.4.26. Addn. to B.P. 250,985; B., 1927, 914).—The electrolyte consists of a solution of sulphuric acid and sodium or potassium sulphate.

H. ROYAL-DAWSON.

Electrolytic rectifier. LE R. P. BENSING, Assr. to G. P. KOELLIKER (U.S.P. 1,645,085, 11.10.27. Appl., 31.8.25).—An electrolytic rectifier comprises a lead anode, a magnesium or silicon cathode, and an aqueous electrolyte containing 20—25% of potassium fluoride and 1—2% of sodium hydroxide. T. S. WHEELER.

Electrolytic [rectifying] cell. E. ROMANELLI, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,648,963, 15.11.27. Appl., 30.7.26).—The cell comprises a lead anode, a filming cathode of zirconium, and an electrolyte of sulphuric acid, *d* 1.25. T. S. WHEELER.

Impregnation of porous electrodes for primary and secondary cells. K. OETTL (G.P. 444,181, 18.9.25).—High-conductivity electrodes are produced by sucking the impregnating medium into the electrodes by evacuating the air. J. S. G. THOMAS.

Carbon electrode. COMP. LORRAINE DE CHARBONS, LAMPES, ET APPAIREILLAGES ELECTRIQUES (Addn. No. 31,586, 27.10.25, to F.P. 594,697; B., 1926, 284).—Catalysts used in accordance with the prior patent are employed either in acid or acid electrolytes, but not in both. For use in alkaline electrolytes, salts of cerium and manganese, borates, resinsates, oleates, etc. may be employed as catalysts, whilst in acid electrolytes, compounds of copper, iron, etc. may be similarly used. J. S. G. THOMAS.

Manufacture of preformed positive electrode. R. C. BENNER and J. H. FINK, Assrs. to PREST-O-LITE STORAGE BATTERY CORP. (U.S.P. 1,654,778, 3.1.28. Appl., 10.7.23).—Finely-divided, electrolytically-deposited lead is oxidised electrolytically until 50—90% is converted

into the peroxide, and the product is applied to a support. J. S. G. THOMAS.

Manufacture of preformed negative electrodes. R. C. BENNER, A. J. BRIGGS, and J. H. FINK, Assrs. to PREST-O-LITE STORAGE BATTERY CORP. (U.S.P. 1,654,779, 3.1.28. Appl., 10.7.23).—Finely-divided lead is prepared and applied to a support under conditions substantially preventing oxidation. J. S. G. THOMAS.

Manufacture of electron-emitting bodies [cathodes]. WESTINGHOUSE LAMP Co., Assees. of W. C. SPROESSER (B.P. 281,687, 1.12.27. U.S., 1.12.26).—A fabricated metal core, *e.g.*, of tungsten wire, is impregnated with activating metal, *e.g.*, thorium, by embedding in the latter and heating in a vacuum or inert atmosphere. J. S. G. THOMAS.

Resistance element. R. F. BUCHHOLZ, Assr. to ATWATER KENT MANUF. Co. (U.S.P. 1,656,901, 24.1.28. Appl., 24.6.24).—Lampblack is distributed through a mixture of litharge and glycerin in such proportion that the specific resistance of the product is 0.1—2 megohms/c.c. H. ROYAL-DAWSON.

Apparatus for the electrometric determination of hydrogen-ion concentration. M. TRÉNEL (U.S.P. 1,675,421, 24.1.28. Appl., 19.11.24. Ger., 6.12.23).—See G.P. 399,410; B., 1926, 208.

Manufacture of transparent substances from non-metallic and non-transparent materials [oxides]. F. SKAUPY, H. HOFFMANN, and H. SCHMIDT, Assrs. to GEN. ELECTRIC Co. (U.S.P. 1,658,659, 7.2.28. Appl., 5.10.22. Ger., 23.2.23).—See B.P. 211,824; B., 1924, 598.

Resistance material. T. A. F. HOLMGREN (U.S.P. 1,658,334, 7.2.28. Appl., 14.1.25. Swed., 22.1.24).—See B.P. 228,142; B., 1926, 98.

Employment of self-baking electrodes. J. WESTLY, Assr. to NORSKE AKTIESELSKAB FOR ELEKTROKEM. IND. OF NORWAY (U.S.P. 1,657,948, 31.1.28. Appl., 5.1.25. Norw., 17.1.24).—See B.P. 227,822; B., 1925, 813.

Metallic [mercury] vapour lamp. R. L. M. BELLEAUD and J. BARROILLIER (U.S.P. 1,656,898, 24.1.28. Appl., 30.1.24. Fr., 14.2.23).—See B.P. 211,455; B., 1924, 860.

Controlling chemical reactions (B.P. 282,168 and 282,170).—See I. **Resistant alloy** (B.P. 283,354). **Nickel sheets** (B.P. 275,221). **Electrodeposition of metals** (B.P. 283,288).—See X. **Insulating material** (U.S.P. 1,656,737).—See XIV.

XII.—FATS; OILS; WAXES.

Acid soaps. M. HARTMANN and H. KÄGI (Z. angew. Chem., 1928, 41, 127—130; cf. A., 1925, i, 1045; B., 1925, 378).—The higher fatty acid *N*-carboxylic esters of asymmetrically-substituted ethylenediamines, *e.g.*, diethylaminoethyloleamide ("sapamine"), yield readily soluble neutral salts with most inorganic and organic acids possessing the properties of soaps. The bases are readily obtained by heating the fatty acid or its chloride with diethylethylenediamine, and may be distilled in a high vacuum. The salts are viscous liquids in the anhydrous condition, and their aqueous or acid solutions

form a lather even at great dilution, the limit being at 0.5 pt. per million. Addition of alkali causes the base to be precipitated and destroys the lathering power, whilst the salt is precipitated in a colloidal hydrated form from concentrated solution on addition of sodium chloride. Unlike alkali soaps, these salts are not precipitated by metallic salts in dilute solution, since, when precipitation would normally occur, the insoluble metallic salt remains in colloidal solution; thus, on adding silver nitrate to the hydrochloride of the base colloidal silver chloride, which is not coagulated by nitric acid, is formed. Colloidal solutions of gold and other metals may be readily obtained by this method. The aqueous solutions of the salts have remarkable oil-emulsifying properties, the surface tension at the oil-aqueous solution interface being too small to measure by known methods. This property makes the solutions of value in the textile and dyeing industries, as in their presence, even in minute quantity, vegetable and animal fibres are very readily wetted. In certain cases where sensitivity to alkali is an objection, the base is converted into a quaternary ammonium base, stable to alkali, by heating with methyl chloride, dimethyl sulphate, or a similar substance. **W. J. POWELL.**

Lathering power of mixed sodium soap. Y. KAWAKAMI (J. Soc. Chem. Ind. Japan, 1927, 30, 216—221).—The froth value of the soap solution is measured as quickly as possible after vigorous shaking (usually after 1 min.), and the lathering power is classified into three grades, according as the froth value is above 80, between 80 and 50, and below 50. If the specific volume of froth, i.e., (froth volume/froth value) \times 100, is less than 5, after being kept for 5 min., the lathering power is regarded as moderate, even when the froth value is above 80. Thus, the lathering power of the three-component system sodium palmitate-sodium oleate-sodium laurate has been measured, the results being shown in the form of three-dimensional graphs. From the results obtained it is possible to anticipate the lathering power at 20° and 40° of tallow, coconut oil, and olive oil soaps, and the limit of good lathering composition of mixed soap consisting of tallow and coconut oils. **Y. NAGAI.**

Apparatus for the automatic recording of numbers of extractions in the quantitative analysis of fats and the like with the Soxhlet apparatus. G. RANKOW (Z. angew. Chem., 1927, 40, 1559—1560).—In order to diminish the amount of supervision required in the use of the Soxhlet extractor, a device has been arranged to record the number of extractions actually performed. It consists of a glass or metal cylindrical float in the extraction portion of the Soxhlet suspended from a balance beam, on the other side of which is a metal rod. Each time the extractor fills up an electrical contact is made which operates the recording apparatus and rings a bell. As for any seed etc. a definite number of extractions are necessary for completeness, the process can by this means be somewhat shortened without loss of accuracy. **C. IRWIN.**

Apparatus for the determination of fatty materials. S. Uxo (J. Soc. Chem. Ind. Japan, 1927,

30, 428—429).—A modification of Soxhlet's extraction apparatus. **Y. NAGAI.**

Recent advances in the hydrogenation of oils. E. R. BOLTON (J.S.C.I., 1927, 46, 444—446 T).—The older methods employing powder catalysts are compared with a new continuous process using a fixed catalyst, which is regenerated repeatedly without loss. The advantages of the continuous hydrogenation process—such as the avoidance of the necessity for filtration of hydrogenated oil—are emphasised, and data given showing the ease of control of the degree of hydrogenation. The composition of oils hydrogenated by different processes is discussed, and it is shown how composition can be altered at will by varying the methods of operating the continuous process.

Selective hydrogenation. K. A. WILLIAMS (J.S.C.I., 1927, 46, 446—448 T).—A method is described by which the extent of the selective effect in the hydrogenation of oils may be measured. The method was applied to the hydrogenation of a number of oils under varying conditions. It was shown that the degree to which the reaction is selective is inversely proportional to the iodine value of the oil treated, and, for a particular oil, directly proportional to the temperature of reaction. With fatty acids the selective effect is directly proportional to the iodine value of the original material.

M.p. of hydrogenated oils. K. A. WILLIAMS (J.S.C.I., 1927, 46, 448—449 T).—The results are given of measurements of the melting points of cottonseed oil hydrogenated, under varying conditions, to different extents. A relation was deduced between the melting point of the hardened product, the temperature of hydrogenation, and the corresponding extent of selection in the reaction.

Hydrogenation of fatty acids and of mixtures of fatty acids with neutral oils. R. G. PELLY (J.S.C.I., 1927, 46, 449—454 T).—In a whale oil containing 12% of free fatty acids the free fatty acids were more fully hydrogenated than the neutral oil. Hydrogenation of neutral oils and fatty acids and of known mixtures with activated nickel turnings or with nickel carbonate-kieselguhr catalyst, followed by examination of the separated neutral oil and fatty acids, showed that (1) the rate of hydrogenation of fatty acids alone is less than that of neutral oil alone; (2) when mixtures of neutral oil and fatty acids are hydrogenated, the rate of hydrogenation of the acid is increased—sometimes beyond the rate of neutral oil alone—while the rate of hydrogenation of neutral oil is greatly retarded; (3) the hydrogenation of neutral glycerides is retarded even by saturated fatty acids. It seems evident that the carboxyl group of the fatty acid molecule is the factor of chief importance.

Acid values of fats and oils. Method of determining the barium values of fats and oils. W. L. DAVIES (J.S.C.I., 1928, 47, 24—26 T).—In titrating the free fatty acids in fats and oils, it is suggested that either alkaline-earth bases should be used as titrating liquids or that excess of neutral alkaline-earth salt solution should be added before titrating with caustic soda or potash, both methods being carried out in alcoholic

solution. For coloured fats in ethereal solution, the end-points can be observed in an aqueous layer underneath containing excess alkaline-earth salt. The insoluble barium value can be determined by ascertaining the amount of neutral barium chloride solution necessary to overcome totally hydrolysis of the saponified fat in dilute aqueous solution, the addition of successive volumes of standard barium chloride generating acidity which can be accurately titrated with alkali. The points obtained mostly lie on two straight lines, the equivalent of the barium chloride value of the point of intersection being the insoluble barium value. This has been determined for a number of fats, and inferences from such data are drawn. The conditions of titrating free alkali when determining the saponification number of a fat are investigated.

Test for refined olive oils in virgin olive oils.

A. BAUD and COURTOIS (Ann. Falsif., 1927, 20, 574—577).—When examined by ultra-violet light filtered through a Wood screen, refined olive oils show a characteristic tint and fluorescence which serves to differentiate them from virgin oils. With transmitted light virgin oils show a yellow-brown colour without fluorescence; refined oils have a blue tint. In very thin films the yellowish colour of the former is not apparent, but the latter show an intensified blue fluorescence. By reflected light the blue coloration is masked by a milky fluorescence.

J. R. NICHOLLS.

Catalytic decomposition of soya bean oil by active carbon. S. HAGA (J. Soc. Chem. Ind. Japan, 1927, 30, 618—624).—Soya bean oil was decomposed by heating with Merck's animal charcoal as catalyst. The cracked oil produced was yellow, had d 0.812, acid value 11.5, n_D 1.4551, and was composed of gasoline, 14.7—23% (containing 71.7% of saturated hydrocarbons, 3.7% of aromatic hydrocarbons, and 24.6% of unsaturated, non-benzenoid hydrocarbons); kerosene, 34.7—38% (containing 72% of paraffin hydrocarbons, 5% of aromatic hydrocarbons, and 22% of unsaturated, non-benzenoid hydrocarbons); and neutral oil, 38—46.6%.

Y. TOMODA.

Examination in Wood's light of some varieties of cod-liver oils. H. MARCELET (Compt. rend., 1928, 186, 226—228).—Twenty-four samples of cod-liver oil, representing varieties used in pharmacy and industry, were examined under Wood's light (3340—3906 Å.) and were found to give a fluorescence which is not destroyed by heating at 150—200°, and which varies from very pale yellow to brown or golden yellow according to the quality of the oil. On account of the difficulty of obtaining the special quartz crucibles most suitable for use as containing vessels, the author places a drop of oil on a sheet of non-fluorescent glass or paper, and examines the streak from the falling drop when the sheet is placed vertically 10 cm. in front of a cardboard screen coated with a non-fluorescent animal charcoal. The drops themselves may appear chestnut-brown in such cases.

J. GRANT.

Lower glycerides of palmitic acid. W. BRASH (J.S.C.I., 1927, 46, 481—482 r).—The lower glycerides of palmitic acid were prepared by heating lead palmitate with the corresponding glycerol chlorohydrin. It was

found impossible to prepare the β -monochlorohydrin in a satisfactorily pure condition. The melting points of the glycerides obtained are: α -monopalmitin 68°, $\alpha\gamma$ -dipalmitin 61.5°, $\alpha\beta$ -dipalmitin 68.5°.

Fatty acids of cohune nut fat. T. P. HILDITCH and N. L. VIDYARTHI (J.S.C.I., 1928, 47, 35—37 r).—Cohune nuts from British Honduras consisted of outer fibrous layer 18.1%, nut (shell) 70.0%, and (kernel) 11.9%; the fat content of the kernel varied from 48—55% and formed a soft, faintly cream-coloured fat (acid value 0.9, sap. value 251.0, iodine value 9.8, unsap. 0.47%; mixed fatty acids: sap. value 268.0, iodine value 10.4, titer 21.2°). After refining and deodorisation the fat was comparable with, but not quite equal to, a high-class coconut fat; there appears to be no reason why cohune nut fat should not be employed for the same purposes as the latter fat; if adequate attention is paid to the collection and transport of mature fruit under hygienic conditions. The mixed fatty acids from the fat were converted into methyl esters and fractionated, and from the results it was estimated that the following acids were present: caprylic 7.5%, capric 6.5%, lauric 46.5%, myristic 16%, palmitic 9.5%, stearic 3%, oleic 10%, and linoleic 1%. The fat is thus very closely similar to coconut fat in its general composition.

Fatty acids of shark- and ray-liver oils. IV. **Fatty acids of Kokonohoshi-Ginzame liver oil.** Y. TOYAMA and T. TSUCHIYA (J. Soc. Chem. Ind. Japan, 1927, 30, 116—122).—The authors have examined the fatty acids of the liver oil from Kokonohoshi-Ginzame (*Chimaera barbouri*, Garman), the sample being the same as that used previously (cf. Toyama, B., 1924, 431, 719). This oil, like Yamato-torpedo liver oil, is remarkable for its high content of unsaponifiable matter (32.99% of the oil), which consists essentially of alcohols of the selachyl alcohol group. The fatty acids contain more than about 10% of saturated acids. The unsaturated acids consist for the most part of the acids of the oleic series; the acids more unsaturated than those of the oleic series are present in extremely small quantities only. The saturated acids consist preponderantly of palmitic acid with small quantities of stearic, arachidic, and behenic acids, and an acid $C_{24}H_{48}O_2$. The acids of the oleic series consist of zoomaric and oleic acids, an acid $C_{20}H_{38}O_2$, and cetoleic and selacholeic acids. Amongst the acids more unsaturated than those of the oleic series, the acids $C_{20}H_{32}O_2$, $C_{22}H_{34}O_2$, and $C_{22}H_{36}O_2$ seem to form the main constituents.

K. KASHIMA.

Increase in the free fatty acid content in herring oils during preservation, and influence of various substances on the increase. S. UENO and N. KUZEI (J. Soc. Chem. Ind. Japan, 1927, 30, 275—279).—The acid value of 33 samples of different composition was measured from time to time during the course of 367 days. Oil heated at about 100° showed an increase of acid value almost equal to that of the untreated oil, from which it is concluded that the increase of fatty acid observed during storage of the oils is chiefly due to the decomposition of glyceride by oxygen in air and not to bacteria or enzymes. Oils that had been heated with oleates of sodium, potassium, lithium, calcium, copper,

mercury, or zinc showed greater increase in acid value than the original oil. The degree of acceleration of these salts depends on the total amount of added salts. Boric acid, arsenious acid, and animal charcoal had no accelerating action. N. KAMEYAMA.

Acetyl value of Japanese fish oils in the hardened oil industry. S. UENO and K. YASUHARA (J. Soc. Chem. Ind. Japan, 1927, 30, 388—389).—The commercial crude oils give variable results and cannot be taken as characteristic of the marine animal oils. The authors suggest that, in determining the quality of fish oils for the hardened oil industry, the acetyl value should be excluded. Y. NAGAI.

Quality of fish oils from the point of view of the hardened oil industry of Japan. II. Sardine oil. S. UENO and K. YASUHARA (J. Soc. Chem. Ind. Japan, 1927, 30, 348—350).—The characteristics of 75 samples of commercial sardine oils of Japan have been determined. The oils had d_4^{20} 0.927—0.933, n_D^{20} 1.479—1.481, saponif. value 187—197, iodine value (Hübl) 160—190, acetyl value 18—30, Hehner value 95—96, and Reichert-Meißl value less than 1.0. Acid value was irregular, but generally lower than that of the herring oil. It is considered that commercial sardine oils often contain some herring oils. Y. NAGAI.

Composition of the body oil from sperm whale. I. Fatty acids. II. Unsaponifiable matter. Y. TOYAMA (J. Soc. Chem. Ind. Japan, 1927, 30, 519—527, 527—532).—I. The oil prepared exclusively from the body blubber of sperm whale (*Physeter macrocephalus*, L.) deposits at ordinary temperatures large amounts of a crystalline solid having d_4^{20} 0.8806, d_4^{20} 0.8733, n_D^{20} 1.4620, acid value 1.24, saponif. value 131.6, iodine value 82.4, unsaponifiable matter 36.40%, and fatty acids 64.13%. The fatty acids are liquid at ordinary temperature, and have d_4^{20} 0.8918, d_4^{20} 0.8847, n_D^{20} 1.4602, n_D^{20} 1.4564, neutralisation value 199.2, saponif. value 201.8, iodine value 87.4, and ether-insoluble bromides 5.55%. The fatty acids consist of about 10% of saturated and 90% of unsaturated acids chiefly of the oleic series, together with a small proportion of acids still more unsaturated. Myristic, palmitic, stearic, and a small amount of arachidic acids form the saturated acids, palmitic acid being preponderant. Lower saturated acids with less than 14 carbon atoms, probably containing lauric acid together with lower members, are also present in small amount. The acids of the oleic series contain zoomaric and oleic acids, an acid $C_{20}H_{38}O_2$, and cetoleic acid. An acid $C_{14}H_{26}O_2$ is also present in small amount. Lower members of the oleic series with less than 14 carbon atoms are absent or present only in minute amount. The acids more unsaturated than those of the oleic series contain in addition to C_{22} acids also C_{20} and C_{18} acids. The presence of clupanodonic acid, $C_{22}H_{34}O_2$, is confirmed. No evidence is obtained for the presence of physetoleic acid. II. The unsaponifiable matter forms at 20° a crystalline solid, having d_4^{20} 0.8508, d_4^{20} 0.8413, n_D^{20} 1.4500, saponif. value of acetylated product 186.6, iodine value 72.2, and cholesterol content (by digitonin method) 0.44%. It consists chiefly of oleyl alcohol (octadecenol), cetyl alcohol, and octadecanol, of which oleyl alcohol preponderates. It

contains also small amounts of alcohols more unsaturated than oleyl alcohol, among which highly unsaturated alcohols giving ether-insoluble bromides are found. Besides these alcohols, cholesterol is present. Hexadecanol is absent or present in minute amount. Tetradecanol was not detected. Oleyl acetate, b.p. 217—220°/15 mm., yields, on oxidation with potassium permanganate in acetic acid solution, nonoic acid and acetoxynonoic acid; hence the constitution of oleyl alcohol is probably $Me \cdot [CH_2]_7 \cdot CH : CH \cdot [CH_2]_7 \cdot CH_2 \cdot OH$, which is identical with that from liver oil of *Chlamydoselachus anguineus*, Garman (J. Soc. Chem. Ind. Japan, 1923, 26, 37; 1924, 27, 468). K. KASHIMA.

Morning-glory (Asagao) oil. I. Properties of the oil. K. MATSUMOTO (J. Soc. Chem. Ind. Japan, 1927, 30, 205—207).—The black and white seeds of "Asagao" (*Pharbitis Nil.*, Chois.) contain 11.85% and 12.13% of the oil, respectively, which is yellow and has a slightly unpleasant smell. The black seed oil has d_4^{20} 0.9255, n_D^{20} 1.4722, acid value 5.3, saponif. value 191.9, iodine value (Hübl) 94.8, Reichert-Meißl value 0.59, and unsaponifiable matter 2.25%. Those of the white-seed oil differ very slightly from those of the black-seed oil. Both oils solidify at about 0°. By the elaidin test, they become turbid after 2 hrs. and yield solid precipitates; after 24—33 hrs. they become buttery masses. The mixed fatty acid (free from unsaponifiable matter) of the oil is a white solid mass yielding no ether-insoluble compounds by bromination, and has m.p. 33—34.5°, iodine value (Hübl) 104.9, neutralisation value 201.4, mean mol. wt. 278.5. Morning-glory oil is a non-drying oil. Y. NAGAI.

Fuel oil from soya bean oil. SATO with others.—See II. **Activity of a nickel catalyst.** LUSH.—See III. **Coconut oil in butter.** ATKINSON and AZADIAN.—See XIX.

PATENTS.

Preparation of fats of a plastic and lithe consistency. K. ERSLEV (U.S.P. 1,657,380, 24.1.28. Appl., 27.8.26. Holl., 9.5.24).—See B.P. 269,384; B., 1927, 531.

Hydrolysis, and particularly splitting, of oils and fats. P. J. FRYER, ASSR. to CATALPO, LTD. (U.S.P. 1,657,440, 24.1.28. Appl., 9.4.21. U.K., 24.4.20).—See B.P. 166,971; B., 1921, 665 A.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Standardisation of turpentine, "terpenoids," and turpentine substitutes, and a method of estimating their technical qualities. F. HEBLER (Farben-Ztg., 1928, 33, 1106—1108).—Turpentine substitutes other than those (e.g., "terpenoids") that resemble turpentine chemically should at least have similar properties to turpentine from the technological point of view. This is not always the case, but the usual chemical and physical tests do not reveal the differences. The effect on the viscosity and stability of a wax emulsion when small quantities of the solvents under examination are added affords valuable information in this connexion.

S. S. WOOLF.

Facet effects on pigments. H. A. GARDNER (Amer. Paint & Varnish Manufs.' Assoc., Jan., 1928, Circ.

No. 321, 97—117).—It was found that the oil absorptions of pigments are appreciably reduced by coating the pigmentary particles with aluminium stearate or other metallic soaps. The laboratory method of achieving this result was to wet the pigment thoroughly with a 2% solution of aluminium stearate in petroleum spirit, that was subsequently evaporated off. Other attempts to "stearate" pigments and reduce the time of grinding of paints gave somewhat anomalous results; e.g., a flat wall paint made by wetting lithopone with aluminium stearate solution and incorporating oil and drier is much heavier bodied than the corresponding paint made on the same formula but omitting the stearate. "Stearated" paints prepared by the laboratory method, however, show less tendency to settle or to "liver" with acid vehicles. Further discussion is given on the possible use of water as a thinner for wall paints, artificial ageing tests on paint (in bulk), and the effects of various vehicles in causing the granulation of paints containing basic pigments. S. S. WOOLF.

Solvents and plasticisers for cellulose esters. A. NOLL (Farben-Ztg., 1928, 33, 1166—1170).—Details of the composition, nature, and various constants of a large number of modern solvents and plasticisers suitable for use in cellulose lacquers are given. S. S. WOOLF.

Brushing lacquers and their future. H. A. GARDNER (Amer. Paint & Varnish Manufs.' Assoc., Jan., 1928, Circ. No. 322, 119—129).—The possibilities of the adoption of brushing lacquers for general painting purposes, as distinct from minor decoration, are discussed, it being shown that the use of a primer composed of red lead, zinc dust, aluminium powder, and linseed oil will overcome the difficulties inherent in the use of nitro-cellulose lacquers on wood. The opinions of 12 authorities on these and other related questions are quoted. S. S. WOOLF.

Scattering of light by particles. LOWRY and MARSH.—See I. "Demolisation." WISLICENUS.—See II. Zinc oxide. DÖRZBACH.—See VII.

PATENTS.

Manufacture of lithopone. J. E. BOOGE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,657,195, 24.1.28. Appl., 6.4.22. Renewed 26.10.23).—Crude lithopone containing less than 0.1% of combined chlorine is concurrently calcined and agitated, air being excluded.

II. ROYAL-DAWSON.

Manufacture of inks. BRIT. DYESTUFFS CORP., LTD., C. HOLLINS, and E. CHAPMAN (B.P. 279,968, 19.8.26).—By the addition of 0.1% of isopropylated, sulphonated mineral oil (B.P. 274,611; B., 1927, 841), sulphonated mineral oil (B.P. 279,990; B., 1928, 50), sulphonated formaldehyde-naphthalene or formaldehyde-phenol condensation products, or alkyl-naphthalenesulphonic acids, ink is made to flow more freely. C. HOLLINS.

Treatment of fouled enamel-removing solutions. W. H. ALLEN (U.S.P. 1,656,154, 17.1.28. Appl., 18.12.25).—Chlorine is passed into alkaline solutions which have become fouled through use in removing enamel, varnish, etc., and the supernatant liquid is drawn off and causticised with lime.

H. ROYAL-DAWSON.

Utilisation of scrap from products made from blood, casein, and glue hardened with formaldehyde. PFENNIG-SCHUMACHERWERKE G.M.B.H. (G.P. 444,218, 28.4.26).—The material is rendered plastic by treatment with alkaline hydrogen peroxide solution, which oxidises the formaldehyde to formic acid.

L. A. COLES.

Manufacture of condensation products obtained from urea and solid polymerides of formaldehyde. F. E. K. STEPPES, Assr. to H. TRAUN & SÖHNE (U.S.P. 1,658,359, 7.2.28. Appl., 19.8.26. Ger., 4.12.24).—See B.P. 271,264; B., 1927, 564.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

The two coagulations of [rubber] latex. R. AUDUBERT and G. LEJEUNE (Rev. gén. Colloid., 1927, 5, 715—722).—The existence of two zones of flocculation for *Hevea* or *Landolphia* latex with an intervening zone of acidity in which complete coagulation occurs is confirmed using preserved latex. Microscopic investigation of the migration of the particles in an electric field indicates that the electric charge remains negative; with the addition of gelatin or casein, however, the charge of the globules shows a change of sign in the second stable zone. The existence and extent of the intermediate stable zone are also dependent on the concentration of rubber in the latex. D. F. TWISS.

Nature of vulcanisation. III. H. P. STEVENS (J.S.C.I., 1928, 47, 37—43 T).—By vulcanising a sufficiently dilute rubber sol with a limited amount of sulphur chloride so that gelling is delayed and mixing while fluid with a raw rubber sol a mixed vulcanised and raw rubber can be obtained with properties substantially the same as those of a rubber vulcanised to the same extent by sulphur chloride in one operation. Similarly, a series of rubber specimens obtained from mixtures of raw and vulcanised rubber latex behave in the same manner as rubber specimens prepared from vulcanised latex direct. Comparisons are based on swelling in solvents and tensile properties. Load-stretch curves are given. A series of rubber specimens was prepared by incorporating cellulose in a finely divided state by mixing latex and viscose. Increased proportions of cellulose result in decreased swelling in solvents, but the physical effect, although progressive, is not parallel with the vulcanising effect of sulphur. Similar experiments with glue are also recorded. It is concluded that combination of sulphur with rubber is essential to vulcanisation, the physical effect of which is brought about by the deposition of caoutchouc (or polyprene) sulphide in a dispersed state in the rubber. The physical properties of a vulcanised rubber will depend on the degree of dispersion and the shape of the particles, which in their turn are modified by the conditions of vulcanisation.

PATENTS.

Vulcanisation of rubber. ROESSLER & HASSLACHER CHEMICAL Co. (B.P. 265,169, 7.1.27. U.S., 27.1.26).—Compounds of the type $(RO \cdot CS)_2S_x$, where x is greater than 2, are claimed as vulcanisation accelerators. They are made by the action of sulphur chlorides on potassium alkylxanthates, the dichloride giving a trisulphide and the monochloride a tetrasulphide. The preparation

of *thioncarbethoxyl tetrasulphide* ($R = Et, x = 4$), *thioncarbo-n-butoxyl tetrasulphide* and *trisulphide*, all yellow oils, is described. For use as accelerators the thioncarbo-alkoxyl polysulphides may be mixed with an amine, e.g., dibenzylamine, aniline, ethylaniline, whereby crystalline substances are obtained; or the oils themselves may be used.

C. HOLLINS.

Vulcanisation of rubber. SOC. ITAL. PIRELLI (B.P. 266,732, 23.2.27. Italy, 24.2.26).—Vulcanisation is accelerated by the salts of carboxylic acids, e.g., of the homologous series of fatty acids (including phenylacetic acid), with alkali metals other than sodium or lithium.

D. F. TWISS.

Vulcanisation of rubber. H. GÜNZLER and W. ZIESER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,653,502, 20.12.27. Appl., 12.4.27. Ger., 21.5.26).—A mixture of zinc oxide and formamide is claimed as an accelerator.

T. S. WHEELER.

Vulcanisation of rubber. W. CARPMARL. From I. G. FARBENIND. A.-G. (B.P. 282,892, 30.9.26).—Formamide is used as an accelerator of vulcanisation.

D. F. TWISS.

Reclaiming of rubber. G. J. MILLER (U.S.P. 1,657,171, 24.1.28. Appl., 22.12.26).—Old rubber scrap containing cotton is comminuted and heated with a suitable acid solution for the removal of cotton and soluble fillers. After being washed, the rubber is then heated in kerosene oil at 120–150° until the maximum amount of impurities has been removed without dissolution of the rubber. The rubber is then separated, washed with a volatile liquid for the removal of kerosene, and dried.

D. F. TWISS.

Preventing the loss of colour of dyed, unvulcanised rubber. CONTINENTAL-CAOUTCHOUC U. GUTTA-PERCHA Co. (G.P. 445,534, 21.3.25).—The rubber is dyed with solutions of lipoid-soluble dyes or dye bases in mixtures containing acetone, rubber-swelling agents, and suitable quantities of high-boiling fatty acids or their esters, and, if necessary, paraffins in addition.

L. A. COLES.

Production of acid- and alkali-resistant coatings of rubber upon metal objects, with the employment of an intermediate layer of rubber. F. AHRENS (B.P. 283,049, 19.5.27. Ger., 16.4.27).—The metal surface to be covered is first coated with one or more layers of a rubber solution, to which addition of an acid substance, or substances capable of liberating acids, has been made. A hard or soft rubber sheet is then applied and the whole is then vulcanised. The reaction product of an unsaturated oil and sulphur chloride, especially if prepared under increased pressure, is advantageous as the acid-generating substance.

D. F. TWISS.

Production of insulating material [from vulcanised rubber]. A. R. KEMP, Assr. to BELL TELEPHONE LABORATORIES, INC. (U.S.P. 1,656,737, 17.1.28. Appl., 28.2.27).—An insulating material is produced by subjecting a series of mixtures containing rubber and sulphur in different proportions to a temperature above that necessary for vulcanisation and mixing them.

D. F. TWISS.

Treatment of water solutions of rubber. E.

HOPKINSON and W. A. GIBBONS, Assrs. to REVERE RUBBER Co. (Re-issues 16,873–4, 7.2.28, of U.S.P. 1,542,388, 16.6.25).—See B., 1925, 643.

XV.—LEATHER; GLUE.

Evaluation of raw stock [skins]. A. C. ORTHMANN (J. Amer. Leather Chem. Assoc., 1928, 23, 1–3).—Samples of raw stock were cut from different parts and placed in an air-tight container. They were cut into pieces 0.5–1 in. square, with as little loss of adhering dirt, salt, and hair as possible, 100 g. placed in a large jar and shaken for 30 min. with 850 c.c. of water at 21°, then the cover was removed, replaced with a 40-mesh screen to retain the hair, and the wash water decanted. The washing was repeated 9 times over a period of 5 hrs., after which the hide pieces and hair were squeezed as dry as possible and dried to constant weight. Results on a number of skins showed salt 2.25–21.0%, material washed out including sand, dirt, salt, etc. 25.9–69.75%, and actual dry hide (including hair) 32.65–74.1%. Samples from different parts of the skin showed but very small differences in analysis.

D. WOODROFFE.

Deterioration of bookbinding leather. F. P. VEITCH, R. W. FREY, and L. R. LEINBACH (J. Amer. Leather Chem. Assoc., 1928, 23, 9–12).—Samples of ten different leather bindings from books kept in a relatively pure atmosphere and not exposed to artificial light or in a room heated by coal fires were analysed. Six samples contained no free mineral acid even after 100–400 years. The highest acidity was 0.53%, in a leather containing pyrogallol and pyrocatechol tannins. The other leathers were tanned with pyrogallol tannins mainly. The good condition of the bindings is attributed to the absence of appreciable acidity, the predominance of a pyrogallol tannage, and storage in a non-polluted atmosphere.

D. WOODROFFE.

Chemical nature of vegetable tanning. C. SCHIAPARELLI and G. BUSSINO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1927, 5, 343–361).—The results of a series of tanning experiments with extracts containing 2% of tannin and using pieces of hide instead of hide powder show that the process of tanning is largely dependent on the hydrogen-ion concentration of the bath. The proportion of water-soluble constituents increases, and the resistance of the leather to hot water diminishes as the acidity of the tanning liquid increases. When pure tannin is used in 2% concentration and the p_H value of the bath is above 2.5, 100 pts. of dry protein in ox hide unites at the ordinary temperature irreversibly with about 60 pts. of tannin (cf. B., 1926, 716).

T. H. POPE.

Chemistry of combined tanning. C. SCHIAPARELLI and L. CAREGGIO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1927, 5, 362–367).—Experiments with hides support Wood's theory on combined chrome-tannin tanning (B., 1908, 384, 1164), one part (basic) of the protein molecule absorbing about 60% of tannin, which represents the proportion of tannin irreversibly absorbed, on the average, by 100 pts. of dry protein, whereas another part (acid) of the same molecule absorbs irreversibly 6.18 pts. of Cr_2O_3 , which approximates to the proportion (6.76 pts.) required to form the chromium dicolla-

genate obtained by tanning in a bath of basicity 33 (1 mol. of sodium carbonate per 1 mol. of chromium alum). This combined tanning should be the ideal tanning, since by it all the reactivities of the protein molecule are saturated (cf. Gustavson, B., 1927, 534).

T. H. POPE.

Utilisation of poplar bark as tanning material.

G. A. BRAVO (Boll. Uff. Staz. Sperim. Ind. Pelli, 1927, 5, 387—402).—Analysis of the barks of various species of poplar gave the following results (%) for *P. nigra*, *P. alba*, and *P. canadensis*, respectively:—Tanning materials, 5.02, 5.26, 4.34; soluble non-tannins, 3.12, 4.40, 4.16; insoluble substances, 81.30, 78.37, 80.55; water, 10.56, 11.97, 10.95. The wood of *P. nigra* contains too low a proportion of tannins to be used for tanning. Extraction of the bark of *P. nigra* with water at about 95° and concentration of the liquid under reduced pressure gave a dry extract which contained 42.00% of tanning materials, 36.09% of soluble non-tannins, 9.83% of insoluble matter, and 12.08% of water, and dissolved completely in hot water, the solution depositing phlobaphens on cooling. For the decolorisation and solubilisation of this extract, the use of blood, oxalic, lactic, or boric acid, or sodium bisulphite proved unsatisfactory, but treatment with a mixture of sodium bisulphite and aluminium sulphate in definite proportions gave good, and that with sulphite-cellulose still better, results. Leather tanned with the crude poplar bark extract, untreated with clarifying or solubilising agents, has a bright chestnut-yellow colour, quite different from the almost dirty colours obtained with other highly coloured tannin extracts. The extract prepared with the help of sulphite cellulose gives a paler yellow, but not a bright, leather, whilst a brown colour is obtained with the extract solubilised with the synthetic coritan. The leathers are of good quality, low ash content, high strength (3.4—3.7 kg./mm.²), and regular elongation (26—27%), and have a soft feel.

T. H. POPE.

Retarding action of tannins in the atmospheric oxidation of the alkali bisulphites.

A. PONTE (Boll. Uff. Staz. Sperim. Ind. Pelli, 1927, 5, 403—415).—Although alkali bisulphites, even in concentrated solution, are readily oxidised in the air, such oxidation occurs only slowly in presence of tanning materials. In general, the tannin does not combine with or retain the free dissolved sulphur dioxide, which, after vigorous and prolonged aeration of the tanning bath, remains in solution in slight amount, only a small proportion undergoing oxidation during the aeration. The formation of additive compounds of tannin and sulphurous acid is thus excluded, but it seems probable that tannin and an alkali bisulphite unite to give an unstable compound which exists only in solution and is analogous to the bisulphite-aldehyde (or -ketone) compounds. No such additive compounds are formed by tannin and normal sulphites.

T. H. POPE.

Permeability of hide and leather. M. BERGMANN (Collegium, 1927, 572—579).—The water-penetration apparatus consists of a Wolff's bottle containing mercury. The middle aperture in the bottle is fitted with a pressure regulator and overflow tube for the surplus water. Water is forced into one of the side inlets of the

bottle and passes out of the other into a tube, arranged above a clamp containing the leather. A mercury manometer is fitted to this tube. The water is forced through the leather or hide and is collected in a measuring cylinder below. The apparatus for measuring the permeability to gas is similar except that the gas forced through the leather is collected over water. Samples of dried, lightly salted, and dry-salted hide, respectively, were soaked for 24 hrs. and tested for water permeability. The results are expressed as the volume of water which was forced through per hour. Tests were made on similar samples which had been kept for 8 weeks, then soaked, limed, and the swelling reduced with a 2% solution of ammonium chloride. The permeability to water increased by 1.9, 3.0, and 12.7 times, respectively, by liming and deliming dried, lightly salted, and dry-salted hide, respectively. Leather from both flesh and grain sides is equally permeable to gases, but that from the flesh side is much more permeable to water. A piece of leather was split giving a grain split 1.5—2.0 mm. thick and a flesh split 3—3.5 mm. thick. The flesh split was less permeable to water (318 c.c./hr.) from the grain side than the flesh side, and less permeable than the grain split from the grain side (3254 c.c./hr.) even allowing for the difference in thickness. The permeability is affected by retannage. D. WOODROFFE.

Softening of hides. V. CASABURI (Boll. Uff. Staz. Sperim. Ind. Pelli, 1927, 5, 368—373).—A short account is given of a series of experiments on the softening of hide in soft water, water of hardness 50° (French), 0.08% sodium hydroxide solution, 0.12% sodium hydrogen sulphide solution, 0.02*N*-sodium citrate solution, 0.128% potassium thiocyanate solution, 0.128% sodium arsenite solution, and 0.11% sodium chloride solution. The full results are to be published later. T. H. POPE.

Pickling of hides. E. SIMONCINI (Boll. Uff. Staz. Sperim. Ind. Pelli, 1927, 5, 383—386).—Experiments on the pickling of hides in baths containing up to 2% of sulphuric acid on the weight of hide and a proportionate amount of salt, with a maximum of 25% of the weight of hide, show that the acid is fixed in a comparatively short time, and that, for equal volumes of bath, the quantity of acid fixed in a given time is proportional inversely to the ratio between acid and hide, and directly to the initial concentration of the bath. If the concentration of the acid and the duration of the action are kept constant, the percentage of acid fixed is not altered by varying the concentration of salt in the bath. The principal effect of pickling is to be attributed to the acid, which combines with and stabilises the collagen fibres, these contracting just as in presence of other tanning agents. The salt acts indirectly by impeding dissociation of the hide-acid compound. T. H. POPE.

Leather substitute. R. DITMAR (Gummi-Ztg., 1928, 42, 913).—Finely-ground sole leather waste (15 g.) was mixed with 45 g. of rape oil, 1.5 g. of castor oil, 6 g. of mineral oil, and 15 g. of sulphur dichloride. A reaction began which was complete in 4 min. and gave a moist dark product; incorporation of this with crêpe rubber and sulphur and then vulcanising for 2 hrs. in the press at 150° gave a final product which is not as good as crêpe rubber vulcanised with sulphur and best

French rubber substitute. Better products have been obtained by using 50 g. of rape oil, 16.5 g. of sulphur, and 14.7 g. of leather. A product manufactured by "Georg Grandel," Augsburg, has the following composition:—rape oil 77.34%, sulphur 12.66%, and ground leather 10%, or 30 pts. of pure "substitute" with 14% of sulphur and 10 pts. of ground leather meal. It had d_{4}^{20} 1.046, acetone extract 19.2%, no free sulphur. The acetone extract is hardly distinguishable from that of "substitute" prepared from pure rape oil.

D. WOODROFFE.

Removal of mineral substances from gelatin by electrodialysis. S. OKA (J. Soc. Chem. Ind. Japan, 1927, 30, 396—402).—After applying a *P.D.* of 50, 100, or 200 volts to the 3% or 10% gelatin solution for from 7 to 15 hrs., the ash contents of the dried substances decreased from 3.50% to 0.05—0.23%. The progress of demineralisation was followed by the measurement of the rise of the electric conductivity of the distilled water put in each electrode compartment. It was found that the velocity of demineralisation was very great at the beginning and rapidly diminished to a very small and almost constant value. The quantity of the cation removed was found to be far greater than that of the anion. Calcium carbonate was found to be the main constituent of the ash before the treatment, but after the treatment ferric oxide predominated. N. KAMEYAMA.

PATENTS.

Manufacture of tanning agents. I. G. FARBEININD. A.-G. (B.P. 266,697, 10.2.27. Ger., 23.2.26).—By using a sulphonating agent, such as chlorosulphonic acid or oleum, which combines with the water liberated, salicylic esters may be sulphonated at 140—190° without appreciable hydrolysis, tanning agents being produced. Examples are phenyl or β -naphthyl salicylate, acetyl-salicylic acid, salicylides obtained from *o*-hydroxycarboxylic acids and thionyl chloride, phosphorylsalicylic acid, and the compounds described in B.P. 218,316 and 252,694 (B., 1925, 181; 1927, 373). C. HOLLINS.

Manufacture of chamois-tanned leather. A. KEMMLER (U.S.P. 1,657,800, 31.1.28. Appl., 28.9.26. Ger., 29.8.24).—See B.P. 266,622; B., 1927, 306.

[Conveyor for] treating hides, skins, and leathers. MASCHINENFABR. TURNER A.-G. (B.P. 277,349, 8.9.27. Ger., 11.9.26).

XVI.—AGRICULTURE.

Influence of superphosphate on the reaction of the soil. C. ANTONIANI (Atti R. Accad. Lincei, 1927, [vi], 6, 319—325).—When applied to neutral, or almost neutral, naked soils free from lime, at the rate of 240—480 lb./acre, superphosphate produces a gradual acidification, but never causes any marked acidity. The fact, noted by other investigators, that superphosphate renders soils alkaline, is doubtless due to the influence of a growing crop or to other factors. T. H. POPE.

Degree of acidity of Norwegian soils. K. O. BJØRLYKKE (Tids. norske landbruk, 1926; Proc. Internat. Soc. Soil Sci., 1927—8, 3, 36).—Examination of a large number of Norwegian soils revealed the following general relationships. Surface soils generally have lower p_H values than subsoils. Acidity is more pronounced in

the older and more completely weathered soils. Soils rich in colloidal matter are usually less acid than sandy soils, and cultivated soils than uncultivated.

A. G. POLLARD.

PATENTS.

Manufacture of mixed phosphatic fertilisers, and recovery of ammonia from industrial gases. H. HOLLINGS, and GAS LIGHT & COKE Co. (B.P. 282,927, 19.10.26).—An industrial gas containing ammonia, *e.g.*, coal gas which has been stripped of its tar, is passed into an absorber charged with superphosphate of lime, which may be mixed with peat, sewage sludge, or other distributing material, until no more ammonia is taken up. The loosely combined excess ammonia is then expelled from the saturated material by passing through it a current of neutral flue gases. A stable mixed fertiliser of constant composition is thereby manufactured, and the gas at the same time is purified from ammonia. It is advantageous to carry out the absorption at about 40° to minimise any condensation of water, naphthalene, or hydrocyanic acid. A convenient cyclic method of carrying out the process, utilising 3 or 4 absorbers, is described.

A. B. MANNING.

XVII.—SUGARS; STARCHES; GUMS.

Volumetric determination of lactose in presence of sucrose. J. H. LANE and L. EYNON (J.S.C.I., 1927, 46, 434—435 T).—In the analysis of sweetened condensed milk the effect of sucrose on the determination of lactose by the authors' method (*ibid.*, 1923, 42, 32 T), can be allowed for by adding certain volume corrections to the amount of sugar solution required by 10 c.c. or 25 c.c. of Fehling's solution. These corrections are given for sucrose/lactose ratios of 3/1 and 6/1, and for any such ratios up to 10/1 the corresponding corrections can be estimated from these data with sufficient accuracy by inspection.

Caramel. H. DRAKE-LAW (J.S.C.I., 1927, 46, 428 T).—Experiments were made on highly coloured caramel with special reference to the dextrose content. The latter is of importance as the fermenting power of caramel and the physiological action on diabetic patients is approximately proportional to the sugar present. A method of analysis based on Fehling's reduction method gives results which are too high, a contention which is established by converting the sugars present into the osazones. The discrepancy in the two methods of testing is in the proportion of 20% to 5%. Highly coloured caramels are therefore suitable for admixture in foods used for diabetic patients and generally as a non-fermenting foodstuff colour.

Adhesiveness of rice, maize, and wheat starches in comparison with potato starch. E. PAROW, A. STIRNUS, and W. EKHard (Z. Spiritusind., 1928, 51, 23—24).—Samples of starches were examined by the Saare test, pastes being prepared by the usual method followed by heating for varying periods on a water-bath. Rice starch prepared by the alkali process and containing a trace of alkali and some protein showed slightly decreasing Saare values with increased periods of heating. Neutralised rice starch yielded values 3—4 times as great and which increased with the heating period.

Rice starch prepared without alkali showed intermediate values and only slight changes with the heating period. The values for maize starch were generally higher with relatively large increases as the heating continued. For wheat starch the values were intermediate between those of maize and rice and varied irregularly with the heating period. Potato starches varied considerably according to their origin, values generally being below those of cereal starches and tending to decrease as the heating period was prolonged. A. G. POLLARD.

PATENTS.

Clarification of cane-sugar juice. J. V. N. DORR, Assr. to DORR Co. (U.S.P. 1,655,049, 3.1.28. Appl., 8.9.24).—After defecation of the rich juice from the sugar cane, the mud so obtained is defecated with thin juice, and the mud from the latter operation is then separated by a filter press. The filtrate and washings are kept separate, the former being returned to the clarifier and the latter passing to the megass for maceration. The cake is flushed from the filter press with water, and the resultant mud is returned to the megass preceding the last mill. F. R. ENNOS.

Treatment [clarification] of cane juice settlings. J. F. BORDEN, Assr. to OLIVER CONTINUOUS FILTER Co. (U.S.P. 1,653,491, 20.12.27. Appl., 22.9.26).—The liquid is treated at 80° with lime to p_H 8.5 and with phosphoric acid to p_H 6—6.9. T. S. WHEELER.

Economical reduction of composite sugar-bearing solutions. H. J. CREIGHTON, Assr. to ATLAS POWDER Co. (U.S.P. 1,653,004, 20.12.27. Appl., 26.3.26).—The mixture of polyhydric alcohols obtained as described in U.S.P. 1,612,361 (B., 1927, 234) is nitrated without separation of mannitol. T. S. WHEELER.

Manufacture of starch. G. M. MOFFETT, Assr. to INTERNAT. PATENTS DEVELOPMENT Co. (U.S.P. 1,655,395, 3.1.28. Appl., 3.3.27).—After steeping the maize in water containing sulphur dioxide, it is washed with water from a preceding separating operation, and then disintegrated. The germ, bran, and fibre are removed in the coarse and fine sloop separations, and the starch is tumbled to free it from gluten and water. It is then filtered and washed twice with fresh water to remove solubles. Economy in the use of water is effected by using that from the starch tables partly for steeping and partly for the germ and coarse sloop separations, and by employing the first and second washings from the starch filters for the fine sloop separation after heating, and for flushing the starch from the tables, respectively. F. R. ENNOS.

Maltose product and production of same. H. C. GORE, Assr. to FLEISCHMANN Co. (U.S.P. 1,657,079, 24.1.28, Appl., 30.3.25. Austral., 28.11.24).—See B.P. 249,428; B., 1926, 458.

Apparatus for diffusing sugar beet or similar substances. J. & F. HOWARD, LTD., and G. E. ROWLAND (B.P. 281,813, 7.10.26).

XVIII.—FERMENTATION INDUSTRIES.

Determination of antiseptic power of hops. T. K. WALKER (J. Inst. Brew., 1928, 34, 61—64).—In the chemical method for the determination of the anti-

septic power of hops, the percentages of α - and β -resins in a hop are determined gravimetrically, and the total preservative power is calculated from the formula $\alpha + \beta/3$, which differs slightly from the formula $\alpha + \beta/4 \cdot 3$ as given by Ford and Tait (cf. B., 1926, 170). Chapman's biological method gives results which agree well with those obtained from the measurement by titration of the amounts of acid formed by *B. Delbrückii* under certain specified conditions in the presence of decoctions of hops. The gradings of hops with respect to their antiseptic powers according to all three methods are in exceedingly close agreement. C. RANKEN.

Relation between time and temperature of storing and their combined influence on fermentation, increases and acid production in varied length of keeping of beer yeast under water at different temperatures. F. STOCKHAUSEN and F. WINDISCH (Woch. Brau., 1928, 45, 31—37, 49—57).—Bottom-fermentation yeasts kept under water at 0° for periods up to 5 days show a slight uniform increase, proportional to the time, of fermentative, reproductive, and acid-forming power, when compared with yeasts similarly stored at 8°. Storage at 20° markedly decreases these characters, the relative amount of such decrease, in the case of fermentation and acid production, increasing with increasing time up to 4 or 5 days. The application of this to yeast conservation and fermentation control is discussed. F. E. DAY.

Examination of grape juice (must) and improvement of wines. D. SCHENK (Pharm. Ztg., 1927, 72, 1409—1411).—Grape juices and wines are improved by adjusting the sugar and acid contents. Sugaring is carried out by adding cane sugar alone or in aqueous solution, care being taken that the alcohol content produced on subsequent fermentation shall not exceed the desired limit. During fermentation, the acidity usually falls owing to the precipitation of tartrates in the lees and the bacterial decomposition of malic acid to lactic acid. The sugar content is determined by multiplying the sp. gr. of the must expressed in Öchsle degrees at 15° by 0.225. The acidity, calculated as tartaric acid, is determined by titration with sodium hydroxide solution using litmus paper as external indicator. If fermentation has commenced, the Öchsle value for the original grape juice must be found by multiplying the alcohol content by 10 and adding that value to the actual Öchsle value. In the improving process the natural deficiency of sugar is made up and any excess acidity corrected by suitable dilution. The number of Öchsle degrees through which the juice must be raised in the process is called the "elevation value." If sugar alone is used, 0.24 kg. is required per degree in the elevation value per 100 litres of juice, but allowance must be made for the volume change on dissolution of the sugar (1 kg. increases the volume by 600 c.c.). If syrup is used 0.22 kg. is required per degree per 100 litres of juice. When the mash is in full fermentation, the latter should be allowed to finish before tests are made. Excess acid may be removed by means of pure precipitated calcium carbonate, but the latter should not be used in quantities greater than 132 g. per 100 litres, equivalent to 2 pts. per thousand of acid,

otherwise a bad taste will develop. Wine may be clarified by the correct addition of pure potassium ferrocyanide.

S. COFFEY.

Composition of crude fusel oil according to different methods. B. LAMPE (Z. Spiritusind., 1928, 51, 30—31).—The content of amyl alcohol in crude fusel oil is not correctly indicated by the official method in which 100 c.c. of oil are subjected to simple distillation and the volumes of the fractions obtained up to 100°, 120°, and 130° determined. The use of a double rectifier of high dephlegmating power and the collection of the fraction which distils from 127° to 132° is suggested as a means of more accurate evaluation. In a comparison of six oils by the two methods, the oil which gives the greatest distillate of b.p. 127—132°, by the method of double rectification, does not yield by the official method the distillate of 90 c.c. which is fixed as the minimum amount to be obtained up to 130°. Accordingly, what would be considered as an oil of the highest value by the first method would be classed by the official method as of inferior quality. The difference between the results depends upon the composition of the amyl alcohol-ethyl alcohol-water mixture which governs the tendency of the amyl alcohol in simple distillation to pass over among the first fractions. If these are treated with water and sodium chloride two layers form, from the upper of which appreciable quantities of amyl alcohol can be distilled.

C. RANKEN.

PATENTS.

Preparing a preservable yeast preparation. E. I. LEVIN (B.P. 271,883, 25.5.27. Swed., 26.5.26).—Pressed yeast is heated for approximately 3 hrs. at 54—55°, and the product, which is spread in a thin layer, with or without admixture of a sterile dry substance, is treated with sterilised pure oxygen at 30° for from 20 to 40 hrs. Subsequent repeated heating at 50—60° for 1—2 hrs. destroys the remaining yeast fungi, leaving unaffected the yeast which has been converted into ascospores, and which can be successfully stored for long periods.

C. RANKEN.

Acceleration of fermentation processes. E. BREDO-WEUSTENRAAD (F.P. 621,439, 24.7.26. Belg., 1.9.25 and 11.2.26).—The maturing of must and beer is accelerated by bringing it in contact with air in open or closed troughs as it leaves the heating vessels.

L. A. COLES.

Utilisation of alcoholic fermentation in order to form large quantities of glycerin by use of sulphurous acid. E. A. BARBET (B.P. 282,917, 13.10.26).—In place of the customary large dose of reducing reagent such as sodium sulphite, sulphurous acid, to which the yeast must be first accustomed, is added to the fermentation medium in very small amount so that its sterilising action remains without effect upon the fermentation. The yield of glycerin from beetroot juice is improved by concentrating the beetroot vinasses without deglycerination and adding to the beetroot juice, as a preliminary to the fermentation, a portion of this concentrate with its high content of organic potassium salts.

C. RANKEN.

Production of sparkling wines. A. CHAUSSEPIED (F.P. 621,341, 5.1.26).—The fermentation process, the addition of liqueurs or other liquids, the saturation with

gas, and the cooling are all effected in the same vessel, means being provided to heat the liquid during fermentation.

L. A. COLES.

Cellulose for butyric fermentation (F.P. 616,424).—See V.

XIX.—FOODS.

Appraisement of contaminated milk. M. FOUASSIER (Ann. Falsif., 1927, 20, 577—580).—Possible sources of contamination are reviewed and a laboratory process is described for removal of insoluble foreign matter by filtration. The interpretation of the results of examination of the sediment is discussed.

J. R. NICHOLLS.

Determination of chlorides in milk. D. RAQUET and A. KERLEVEO (Ann. Falsif., 1927, 20, 580—583).—A criticism of the ordinary methods is followed by a description of two new methods which do not possess the usual disadvantages. (i) To an aqueous extract of the residue from the fat determination by the Adams process 5 c.c. of concentrated nitric acid, 10 c.c. of 0.1*N*-silver nitrate solution, 5 c.c. of 5% potassium permanganate solution, and about 50 c.c. of water are added. After boiling gently for 10 min. and decolorising, 50 c.c. of water are added and the solution is again heated for 10 min. and then cooled. 5 c.c. of nitric acid and 1 c.c. of 0.2*N*-ferric alum are added, and the solution is titrated with 0.1 *N*-ammonium thiocyanate. (ii) To 20 c.c. of milk diluted with about 100 c.c. of water is added 1 c.c. of 15% potassium ferrocyanide solution. The mixture, after addition of 1 c.c. of 30% zinc acetate solution, is diluted to 200 c.c. and filtered. To 150 c.c. of the filtrate are added 5 c.c. of nitric acid and 10 c.c. of 0.1 *N*-silver nitrate. The mixture is again filtered, and the filtrate (150 c.c.) after addition of 1 c.c. of ferric alum solution, is titrated with ammonium thiocyanate.

J. R. NICHOLLS.

Coconut oil in butter. H. ATKINSON and A. AZADIAN (Ann. Falsif., 1927, 20, 593—602).—The methods which have been suggested for determining coconut oil in butter are reviewed, and a method is proposed which consists in obtaining "the insoluble silver number" of the fatty acids soluble in water. The neutralised solution after determination of the Reichert value is slightly acidified by the addition of 2 drops of 0.1*N*-sulphuric acid, 0.1*N*-silver nitrate solution (10 c.c.) is added, and the solution after diluting to 200 c.c. is kept for 15 min. with occasional shaking. The filtered solution (110 c.c.) is titrated by Volhard's method with 0.1*N*-potassium thiocyanate. The difference between the titration and a blank is due to insoluble silver salts, and, after correcting for dilution, is expressed as a percentage of the Reichert value. Any increase of the insoluble silver number suggests admixture of coconut or palm-kernel oils; the addition of animal or other fats with a negligible Reichert value, whilst diminishing this value for the butter, has no appreciable effect on the insoluble silver number. Values so obtained for numerous samples of butter from both cow's and buffalo's milk, with and without the addition of coconut and palm-kernel oils etc., are tabulated.

J. R. NICHOLLS.

Baking value of flour. BRAUN (Ann. Falsif., 1927, 20, 588—592).—A method is described for analysing a

small sample of wheat so that within 30 hrs. all the factors necessary for appraising the baking value of the flour produced from it can be obtained. The wheat (25 g.) is ground in a coffee mill to pass 100-mesh, the larger particles being re-ground until the residue weighs 7.5 g. The sieved product represents a 70% flour. The wet gluten is extracted from 10 g. in order to ascertain its quality and its hydration ratio. Then 1 g. of the flour is treated with 10 c.c. of water, collected on a filter, and washed, the nitrogen being determined on the insoluble material; the value obtained, which corresponds to the gluten nitrogen, when multiplied by 6.25, gives the dry gluten. The gliadin is dissolved by treating 5 g. of the flour with 50 c.c. of 0.5% solution of potassium bicarbonate in 70% alcohol for 24 hrs. After filtering, the gliadin nitrogen is determined on 20 c.c. of the filtrate. The moisture of the flour is determined on 1 g.

J. R. NICHOLLS.

Compounds of ammonia and carbon dioxide (salts of hartshorn); ammonium bicarbonate as a raiser in baking. T. PAUL [with M. LANDAUER and F. KRÜGER] (Z. angew. Chem., 1927, 40, 1539—1548).—Five solid compounds of ammonia, carbon dioxide, and water (exclusive of urea) are known with ammonia content ranging from 21.55% to 43.64%. Increase of ammonia content in aqueous solution results in a decrease of the partial pressure of the two other components, especially of carbon dioxide. In the vapour phase at 100° the compounds are largely dissociated, but molecules of ammonium bicarbonate exist. Baking tests with the different solid salts in the proportion of 7.5 g. of salt to 500 g. of flour showed that with increasing proportions of ammonia the lightness of the product (vol./wt.) decreased. The proportion of residual ammonia in the loaf or cake increased somewhat with the ammonia content of the salt. It was further found that the use of ammonia solution in place of water (without any raiser) gave a denser product than with water alone, and it was concluded that ammonia has an actual adverse effect on the raising process. Ammonium bicarbonate is therefore the best medium for the purpose. "Salts of hartshorn," prepared by the old method of distillation of animal refuse, were found to contain 28—36% NH_3 according to the conditions of resublimation. The double salt $\text{NH}_4\text{HCO}_3 \cdot \text{NH}_2 \cdot \text{CO}_2\text{NH}_4$ and ammonium carbonate are unstable, and trade samples proved to be of irregular composition, whilst the bicarbonate is much more stable. It is therefore claimed that the designation "salts of hartshorn" (Hirschhornsalz) should not be limited to products of high ammonia content, as no definite line can be drawn, and for its principal application the bicarbonate is the most efficient form.

C. IRWIN.

Chemical composition of certain foods. L. SETTIMI (Atti R. Accad. Lincei, 1927, [vi], 6, 315—318).—Results of analysis are given for a number of samples of the following foods, in common use in Italy: tinned meat of various ages, fresh lean beef, cheese, stockfish, lean bacon, and dried mushrooms (*Boletus edulis*).

T. H. POPE.

Nutritive value of the nitrogenous substances obtained from certain kinds of preserved foods.

Experimental researches on white rats. S. BAGLIONI and L. SETTIMI (Atti R. Accad. Lincei, 1927, [vi], 6, 377—383).—The soluble proteins from certain common Italian foodstuffs (cheese, stockfish, dried mushrooms) and the insoluble proteins from cheese, stockfish, and casein not only maintain the nitrogen balance in the organism of white rats, but also effect a storage of nitrogen and an increase in body weight. The insoluble proteins from mushrooms are not able to do this.

O. J. WALKER.

Detection [in sausage] of dyeing with the colouring matter of sandal wood. L. SOEP (Analyst, 1927, 52, 696—699, and Chem. Weekblad, 1927, 24, 624—625).—To detect sandal wood dye in sausage, 50 g. of sausage are boiled with absolute alcohol, the mixture is cooled and filtered, and the filtrate examined spectroscopically. Uncoloured sausage extract shows only very little absorption at 0.59μ , 0.54μ , and 0.44μ , but the coloured extract shows three distinct bands. The lead salt of santalic acid is then precipitated by basic lead acetate, and very small quantities give the precipitate a violet tinge. The precipitate is then collected, washed with hot alcohol, suspended in 50 c.c. of *N*-sulphuric acid, and again collected on a filter. The filtrate shows the three typical bands in the spectroscope. The santalic acid is transferred to ether and the ethereal solution evaporated, the residue dissolved in dilute acetic acid, and tested with hydrogen peroxide for the formation of colourless needles in addition to an amorphous substance, and with potassium nitrite for formation of a brown precipitate and yellow solution. Finally, a confirmatory microscopical examination is made. An alcoholic solution of santalic acid dyes woollen fibre orange to pink in presence of bisulphate, and produces a dirty violet colour on fibre mordanted with ferric chloride with no bisulphate.

D. G. HEWER.

PATENTS.

Sterilising, ageing, and bleaching of flour, meal, etc. E. STAUDT (B.P. 263,760, 9.11.26. Switz., 23.12.25).—The flour etc. is treated with the minimum quantity of halogen oxides, e.g., 1—2 g. of chlorine dioxide to 100 kg. of flour, suitably diluted with an inert gas, powdered silica gel, or a solvent such as water or oil.

F. R. ENNOS.

Extraction of pectin from fruit. H. T. LEO (U.S.P. 1,654,131, 27.12.27. Appl., 7.2.27).—Citrus fruit is subjected to pressure to extract the juice, and the rind, after slicing, is treated with strong alcohol in order to render inactive the enzymes present and to inhibit the action of the acid juices on the pectin. The alcohol is then removed, and the pectin is dissolved out from the rind with a hot weakly acid solution and concentrated to a definite gel standard.

F. R. ENNOS.

Manufacture of powdered or granular jellifying compound. H. T. LEO (U.S.P. 1,655,393, 3.1.28. Appl., 12.7.21. Renewed 6.5.27).—A solution of pectin in boiling water is prepared and, after the addition, preferably first, of the sugar and then of a fruit acid, e.g., tartaric or citric acid, the product is at once dried by atomisation.

F. R. ENNOS.

Apparatus for treating chocolate. BAKER PERKINS, LTD., W. E. PRESCOTT, J. P. BUNCE, and ANC.

ÉTABL. A. SAVY JEANJEAN & CIE. SOC. ANON. (B.P. 283,716, 17.12.26 and 13.6.27).

Method and apparatus for treating crystallised margarine mass. C. HILDEBRANDT-SØRENSEN (B.P. 267,928, 15.3.27. Denm., 16.3.26).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Manufacture of theobromine. E. A. MAUERSBERGER (Chem.-Ztg., 1928, 52, 89—90).—Theobromine is extracted from the waste products of cocoa and chocolate manufacture by either alcoholic or aqueous extraction, the material freed from fat and dried containing 0.8—3.5% of the alkaloid. It is ground, mixed with dry slaked lime, and stirred with water. Combined theobromine is liberated and ammonia and methylamine are evolved. The older method of extraction with 80% alcohol at 60—70° is now uneconomic owing to the excessive loss of solvent involved. The author employs 35% alcohol at 50°, using a filter press in which the cake is washed with water. The washed cake is free from theobromine and contains only 3—8% of alcohol, which is recovered from a dryer. The filtrate is distilled in iron stills until free from alcohol, and then concentrated and neutralised in enamelled pans. Theobromine separates with but little formation of resin. Processes of extraction with hot or cold water involve either concentrating the filtrate until theobromine mixed with albuminous substances separates, or rendering it alkaline and allowing it to settle. A higher filtration pressure is needed, and the wear on filter cloths is greater. Crude theobromine is freed from albumin by converting it into the calcium compound, and from colouring matter by treatment with soda. C. IRWIN.

The DM1 and DM2 indices and the Warsaw Congress. F. DE MYTTENAERE (J. Pharm. Chim., 1928, [viii], 7, 14—25).—The de Myttenaere indices for the toxicities of arsenobenzene preparations were adversely criticised at the Warsaw Congress. Experiments are described showing that the DM index has a precise chemical significance, depending on the amount of arsenobenzene oxidised to arsenobenzene oxide, and that these indices do afford information regarding the composition of commercial arsenobenzene preparations; a case is cited where a sulphoxylate compound of diaminoarsenobenzene contained a considerable quantity of a non-nitrogenous impurity along with some disulphoxylated compound. The DM index very often serves to correct erroneous results obtained for the toxicity of an arsenobenzene preparation by *in vivo* experiments. The latter are not as satisfactory as would be desired. Of two perfectly satisfactory preparations one was toxic to rabbits but non-toxic to mice, whereas with the second the reverse was true.

S. COFFEY.

Determination of impurities in crude camphor.

I. Determination of water and solid matter. S. YAMADA and T. KOSHITAKA (J. Soc. Chem. Ind. Japan, 1927, 30, 356—359).—For the determination of water in crude camphor by distillation with volatile solvents, a device which is a modified form of Normann's apparatus (B., 1925, 512) was used. [It is claimed that adhesion of condensed water on the wall of the still-head is com-

pletely avoided by the use of a still-head provided with a built-in reflux condenser having an inverted part at its end. The use of toluene or xylene instead of benzene is also recommended. More accurate results were obtained than by the usual method of centrifugal separation. Y. TOMODA.

Californian lemon and orange oils. L. BONACCORSI (Boll. Uff. R. Staz. Sperim. Ind. Essenze, 1927, 2, 1—3).—A sample of pale yellow Californian lemon oil had d_{4}^{15} 0.8580, $\alpha^{15} + 63.8^\circ$, n_D^{20} 1.4709, aldehydes (as citral) 2.35%, saponif. value 9.33, fixed residue (water-bath) 2.75% having saponif. value 193.9, solubility in 90% alcohol 1:7. A sample of Californian orange oil gave d_{4}^{15} 0.8518, $\alpha^{15} + 98.5^\circ$, n_D^{20} 1.4693, aldehydes (as citral) 2.62%, saponif. value 4.58, fixed residue (water-bath) 3.46% having saponif. value 160.91, solubility in 90% alcohol (turbid) 1:8.

T. H. POPE.

Constituents of Indian essential oils. XXII. Essential oil from flower heads of *Cymbopogon coloratus*, Stapf. P. P. PILLAY, B. S. RAO, and J. L. SIMONSEN (J.S.C.I., 1928, 47, 52—54 r).—Oil from the flower heads of *C. coloratus*, Stapf, grown in the North Arcot district had d_{20}^{20} 0.9183, n_D^{30} 1.4819, $[\alpha]_D^{30}$ —20.7°, acid value 2.3, sap. val. 39.8, sap. val. after acetylation 111.2, aldehydes (sodium bisulphite) nil. The principal constituents found were *l*-camphene (15%), *l*-limonene (7%), camphor (?) (trace), *l*-borneol (8%), geraniol (10%), sesquiterpene oxide (?) (2—3%), sesquiterpenes (35%), sesquiterpene alcohols (8%). The absence of citral is noteworthy (cf. Bull. Imp. Inst., 1912, 10, 670; Proc. Chem. Soc., 1914, 30, 10).

Characters of essences of aromatic plants cultivated in Calabria. L. BONACCORSI (Boll. Uff. R. Staz. Sperim. Ind. Essenze, 1927, 2, 4—5).—The flower-heads (leaves) of *Majorana hortensis* gave 0.48 (0.43)% of pale yellow essential oil having d_{4}^{15} 0.8912 (0.8868), $\alpha + 7.2^\circ$ (9°), acid value 0.28 (0.56), saponif. value 13.07 (11.20), saponif. value after acetylation 64.72 (73.77), carbonyl compounds (as citral) 1.67 (2.03)%, solubility in 82.5% alcohol 1:2.2 (1:1). The essence of *Thymus citriodorus* gave d_{4}^{15} 0.9020, $\alpha + 3.6^\circ$, acid value 4.48, saponif. value 30.01, carbonyl compounds (as citral) 13.50, phenols trace; soluble in 2 vols. of 70% alcohol. The essence of *Salvia tritobica* gave d_{4}^{15} 0.9200, $\alpha + 10.4^\circ$, acid value 1.68, saponif. value 8.66, saponif. value after acetylation 50.38, carbonyl compounds (as citral) 5.84; soluble in 1 vol. of 80% alcohol. Oil of *Pelargonium radula*, Ait (?) cultivated on the plain (hill) gave d_{4}^{15} 0.8970 (0.8966), $\alpha - 9^\circ$ (—9.2°), n_D^{20} 1.4639 (1.4643), acid value 13.44 (12.30), saponif. value 62.71 (70), saponif. value after acetylation 277.81 (260.32), esters (as geranyl tiglate) 26.43 (29.50)%, total alcohols (as geraniol) 76.40 (72.12), free alcohols 59.20 (52.88), citronellol 36.70 (33.36), solubility in 70% alcohol 1:2.1 (1:2.7).

T. H. POPE.

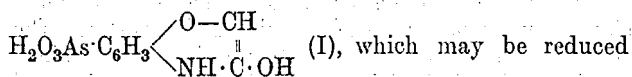
Australian sandal woods and their essences. E. PERROT (Bull. Sci. Pharmacol., 1927, 34, 609—640).—The botanical and histological characteristics of *Santalum spicatum* A. DC. (*Eucarya spicata*) and *Santalum lanceolatum* are described and the chemical and physical

constants of the oils derived from them are compared. From a review of many published values for these constants the following values for the two oils are derived: *S. spicatum*, d_{15}^{20} 0.968—0.972, n_D^{20} 1.498—1.512, $\alpha - 3^\circ$ to -9° , alcohols (as santalol) 90—96%, solubility in 70% alcohol, 1 in 3 to 6 vols.; *S. lanceolatum*, d_{15}^{20} 0.968—0.973, n_D^{20} 1.498—1.512, $\alpha - 30^\circ$ to -40° , alcohols 90—95%, solubility in 70% alcohol, 1 in 3 to 6 vols. Australian sandalwood oil is produced almost exclusively from *S. spicatum*, and in therapeutic action is not inferior to Mysore oil. E. H. SHARPLES.

Zinc oxide for ointments. DÖRZBACH.—See VII.

PATENTS.

Manufacture of organic compounds of arsenic. G. NEWBERRY, and MAY & BAKER, LTD. (B.P. 280,613, 8.7.26).—By the action of suitable reducing agents the arsenic acid derivatives of *o*-nitrophenoxyacetic acids are converted into arsenic acid, arsenoxide, or arseno-derivatives of 3-hydroxybenz-1:4-oxazines. 3-Nitro-4-carboxymethoxyphenylarsinic acid (Christiansen, A., 1922, i, 1203) is reduced by ferrous sulphate and sodium hydroxide to 3-hydroxybenz-1:4-oxazine-6-arsinic acid,



by sodium hyposulphite and magnesium chloride to the 6:6'-arseno compound. The corresponding 8-acetamido derivatives of these are prepared from the 6-acetamidoderivative of (I) obtained by interaction of chloroacetic acid and 2-nitro-6-acetamidophenol-4-arsinic acid (itself prepared by nitration). C. HOLLINS.

Production of aluminium salts of organic acids. H. JUNG (G.P. 444,734, 1.3.25).—Products of therapeutic value as intestinal disinfectants, especially against oxyuriasis, are prepared by treating water-soluble basic aluminium salts of acetic acid or its homologues with aromatic carboxylic acids. *E.g.*, basic aluminium propionate solution, prepared by the action of propionic acid upon a mixture of aluminium sulphate solution and calcium carbonate, is boiled with benzoic acid, when a basic salt with a benzoate:propionate ratio of 1:2 is precipitated, which is collected on a filter, washed, and dried at 100° . L. A. COLES.

Manufacture of a substance producing hypoglycaemia. H. E. DUBIN and H. B. CORBITT, ASSRS. to H. A. METZ LABORATORIES, INC. (U.S.P. 1,653,452, 20.12.27. Appl., 23.8.23).—Cabbage is extracted with alcohol, and the solution formed is treated with charcoal, which is then extracted with glacial acetic acid. The acid is evaporated *in vacuo*, and the residue, dissolved in water, is then treated with dinitrosalicylic acid to precipitate the required product. T. S. WHEELER.

Production of santonin. SOTERIA G.M.B.H., CHEM. PHARM. FABR. (G.P. 444,850, 24.11.22. Addn. to G.P. 346,947; B., 1922, 521 A).—The leaves of indigenous species of *Artemisia*, *e.g.*, *Artemisia monogygna* from the Baltic coast, or *Artemisia maritima*, are used for the production of santonin by the process described in the prior patent. L. A. COLES.

Production of a [stable] colloidal silver-tannin-albumin combination soluble in water. H. COHN and C. STIEBERT (U.S.P. 1,653,087, 20.12.27. Appl., 28.12.26. Ger., 14.3.23).—Diacetyltannin dissolved in sodium carbonate solution is mixed with silver-albumin solution, and the liquid is evaporated *in vacuo* to yield a soluble product of therapeutic value. T. S. WHEELER.

Manufacture of derivatives of organic arsenic compounds. J. PFLEGER and A. ALBERT (U.S.P. 1,653,227, 20.12.27. Appl., 10.6.25. Ger., 19.6.23).—See B.P. 235,864; B., 1925, 738.

Manufacture of alkyl derivatives of cyclotrimethylenearylpirazolones. C. MANNICH (U.S.P. 1,657,544, 31.12.28. Appl., 29.11.26. Ger., 31.12.25).—See B.P. 263,773; B., 1927, 869.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

[Photochemical] bleaching-out paradoxes. LÜPPO-CRAMER (Phot. Ind., 1927, 1099—1100).—It has been shown previously that the photochemical bleaching of pre-exposed silver bromide plates which have been treated with a solution of potassium iodide containing a safranin dye, and the desensitisation of silver iodide plates which have not been pre-exposed, decrease instead of increase with increase in concentration of the dye. Similar results have now been found with un-ripened diapositive silver bromide plates, free from all traces of soluble bromide, and without potassium iodide treatment. In certain cases the densities rise to a value higher than that due to the pre-exposure. The effect is probably due to a peculiarity of the emulsion.

W. CLARK.

Anomalous destruction of nuclei. LÜPPO-CRAMER (Phot. Ind., 1927, 1286—1287).—Bleaching of the latent image with Phenosafranine first increases with dilution of the solution (from 1:20,000 to 1:80,000) and then begins to decrease. It is considered that the bleaching is due to the combined action of the Phenosafranine acting as oxidiser and the bromine remaining adsorbed on the grains and being set free by pre-exposure. There will be an optimum concentration of the dye at which these two results will show a maximum. The dye has a replacing effect on the bromine adsorbed to the grain and the extent of replacement will depend on the dye concentration.

W. CLARK.

Denucleation and desensitisation. LÜPPO-CRAMER (Phot. Korr., 1927, 63, 362—363).—The action of a desensitiser must be on the latent image while it is in the act of being formed and not on the latent image already formed. The actual latent image probably contains silver in a condition too coarse for it to be acted on by the desensitiser. It is possible that the ripening nuclei would be attacked by the desensitisers, and that the action of a desensitiser on a plate treated with chromic acid to remove the ripening nuclei would be less than on a plate not so treated. Experiments show this to be the case.

W. CLARK.

Experiments on hypersensitisation. K. JACOBSON (Phot. Ind., 1928, 56—62).—For the hypersensitisation of kinematograph film, a mixture of Pinacyanol

and Pinaflavol is not altogether satisfactory. If pyridine is added the results are better, but spots are liable to form. Best results are obtained with a mixture of Pinachrome and Pinachrome Violet and ammoniacal silver chloride. By appropriate pre-exposure after sensitisation the sensitivity may be still further increased. Hypersensitised film must be exposed and developed within 48 hrs. of sensitisation. W. CLARK.

Ageing phenomena of sensitised material. A. FUNGER (Phot. Ind., 1927, 1203—1204).—A general discussion of the keeping qualities of sensitive material, with special reference to edge fog. Colour sensitive materials do not keep so well as unsensitised emulsions because no bromide is added to them before coating. W. CLARK.

Sensitometry of reversal emulsions. L. LOBEL and J. LEFÈVRE (Bull. Soc. Franç. Phot., 1927, 14, 288—297).—The sensitometry of emulsions intended for the production of direct positives is considered from first principles. The gamma of the positive curve is equal to that of the negative curve, and is symmetrical to it about a line drawn parallel to the exposure axis through the ordinate $D/2$, where D is the maximum density developable with the emulsion. The position of the positive curve depends on the value of D alone. For correct positive reproduction, the emulsion should have a gamma-infinity equal to unity, and the solarisation density (i.e., the density corresponding to an exposure at which solarisation commences, development being to gamma infinity) must be equal to the maximum density obtainable by complete reduction of all the silver halide. If it is desired to render pure whites, the exposure must be such that the value of I is equal to the exposure value at which solarisation commences. The speed of an emulsion for reversal is higher the thinner the emulsion coating. W. CLARK.

Error in sulphide-toned images. K. KIESER (Phot. Ind., 1927, 1257—1258).—In the ordinary process for sulphide toning, using ferri cyanide and sulphide, care must be taken that all the ferrocyanide and ferri cyanide is washed out of the emulsion before sulphiding. The sulphide will reduce any ferri cyanide to ferrocyanide, and this, on keeping, and especially on exposure to light, decomposes with the formation of blue products which give markings on the prints. W. CLARK.

Tentative hypothesis of the latent image. II. A. P. H. TRIVELLI (Phot. J., 1928, 68, 67—72; cf. B., 1928, 107).—A probable elementary photo-electric cell on the surface of the silver halide grains of an emulsion is pictured, based on the conclusions of Wightman and Quirk (B., 1927, 268), and of Clark (*ibid.* 507), that the sensitising specks of high-speed emulsions as well as the latent image probably consist of both silver and silver sulphide. In the photo-electric system, silver is regarded as the cathode, silver sulphide as the anode, and silver bromide as a solid electrolyte. A mechanism of this cell in producing sensitivity is described. The theory is also applied to the explanation of the action of a flash exposure in increasing desensitisation by chromic acid. On the assumption that optical sensitising is due to an increase of photo-conductivity for longer wave-lengths, a mechanism is given of how dyes or silver sulphide could

act as optical sensitisers, based on the assumption that dye specks or finely-divided silver or silver sulphide lying in the field of the photo-electric cell $\text{Ag—AgBr—Ag}_2\text{S}$, increases its photo-conductivity to long wave-lengths above that of a similar cell without these substances present. W. CLARK.

Photographic density. J. C. VAN SELMS (Dissertation, Utrecht, 1927).—The extinction of a photographic plate is in general independent of the wave-length for ordinary development. With plates strongly exposed and developed for a very short time, the extinction increases with decreasing wave-length. A method is described for measuring in an absolute manner the intensity of light scattered at all angles by a photographic density. Measurements with blue and with red light showed that the intensity of light scattered at different angles is independent of the wave-length. The intensity of scattered light was small at big angles, and very high at small angles, but no relation could be deduced between intensity and angle. A curve is given showing the intensity of the scattered light at different angles as a function of the plate extinction. The intensity of light scattered at a given angle is in certain cases independent of the extinction of the plate. The surface of the plate plays only a small part in determining the scatter. The effect of diffraction round the silver grains was examined on the basis of Rayleigh's law, but the experimental results did not agree with the theoretical. The theory of Mie would probably be more applicable. The intensity of the scattered light could be explained if the variation of the refractive index of the gelatin in the neighbourhood of the silver grain were known. W. CLARK.

Errors produced in slow development with glycine. A. and L. LUMIÈRE and A. SEYEWETZ (Bull. Soc. Franç. Phot., 1927, 14, 306—308).—The observations of Lobel and Lefèvre (B., 1927, 461), that as far as the final result is concerned, the actions of glycine and metol-quinol are identical, is confirmed. In the use of a dilute solution of glycine as a tank developer, however, troubles occur owing to the formation of a film of oxidised developer at the plate surface. This film is invisible and prevents the developer from wetting the plate at the parts of the sensitive surface where it is formed. The trouble can be partly but not entirely eliminated by wetting the emulsion surface before developing. W. CLARK.

Silver content of photographic layers. F. WEIGERT and F. LUHR (Naturwiss., 1927, 15, 788).—The authors have improved the electrometric titration of silver to such an extent that on titration with 0.0001N-potassium iodide it is possible to determine 0.001 mg. of silver in 20 c.c. of solution. Using this method, the amount of free silver in unexposed photographic plates was measured. In four different commercial plates the amount of silver varied from 1.88×10^{-4} to 2.80×10^{-4} mg./cm.² That this is ripening silver is proved by the fact that the amount of free silver increased with the time of ripening, the increase being greatest with silver chloride, less with silver bromide, and nil with silver iodide. When the plate is treated with persulphate, the amount of free silver decreased and the sensitivity

diminished, in accordance with the observations of Clark (B., 1927, 507). Silver sulphide is not attacked by persulphate under the conditions studied. W. CLARK.

Toning with selenium compounds. J. MILBAUER (Chem. Listy, 1927, 21, 509—515).—A large number of selenium compounds is examined from the point of view of their direct or indirect use in toning photographic images. The procedure recommended is the following: Prints or lantern slides are fully developed, fixed, and washed, and then treated in a bath containing 80 c.c. of a 1% solution of crystalline sodium sulphide and 10 c.c. of a 1% solution of sodium selenite or selenious acid. After 20 min. or less in this bath, intense brown tones are obtained with chlorobromide papers, or brown-violet tones with pure bromide papers. W. CLARK.

Fastness to washing of dyed silver bromide. LÜPPO-CRAMER (Phot. Kor., 1927, 63, 193).—Sensitisers adsorbed to silver bromide in photographic plates can be removed by washing. Plates sensitised with Pinaflavol, Rhodamine B, or Isoquinoline Red lose their yellow sensitiveness on washing. With erythrosine there was no decrease, but an increase on prolonged washing, although in this case the undyed plate gave a similar increase after such washing. W. CLARK.

Light-sensitivity of dyes. II. A. STEIGMANN (Kolloid-Z., 1928, 44, 173—175).—Theories of the sensitisation of photochemical reactions by colouring matters are discussed. E. S. HEDGES.

PATENTS.

Obtaining photographic images. E. GAY (B.P. 283,274, 6.10.26. Addn. to B.P. 282,894; B., 1928, 173).—The light-sensitive diazo compounds of the prior patent are mixed with *O*-acyl derivatives of the phenols or naphthols used as coupling components in development. The phenols or naphthols are liberated by development with ammonia gas or a dilute alkaline bath, and a coloured positive image is obtained. Examples of suitable *O*-acyl derivatives are phenyl acetate, α -naphthyl benzenesulphonate, 2-acetamido-8-acetoxynaphthalene-6-sulphonic acid (from γ -acid), 1-acetamido-8-acetoxynaphthalene-3:6-disulphonic acid (from *H*-acid), and 1:8-diacetoxynaphthalene-3:6-disulphonic acid (from chromotrope acid); the last-mentioned gives with 2-diazo-1:4-dimethoxybenzene a violet-red image. C. HOLLINS.

Sensitising photographic films or plates. O. LOISA (G.P. 443,677, 25.1.25).—Sensitising in solutions of basic sensitisers is carried out for 10—30 min. with simultaneous cooling of the bath to below 15°, and drying the bathed material at below this temperature. Correct colour-tone rendering can be obtained without the use of a filter. W. CLARK.

Protective layers for photographic gelatin emulsions. P. G. and C. GLASER (F.P. 617,497, 14.6.26).—The emulsion is coated with a varnish consisting of a solution of celluloid and gum lac, to which is added a mixture of glacial acetic acid, ethyl acetate, and amyl acetate. W. CLARK.

Actinism-proof cellulose ester composition. J. H. CLEWELL, JUN., Assr. to E. I. DU PONT DE NEMOURS &

Co. (U.S.P. 1,647,435, 1.11.27. Appl., 7.1.24).—Transparent sheets of pyroxylin are coated on both sides with a colourless, transparent, oil-resin varnish, e.g., a copal-linseed oil mixture, impervious to ultra-violet light.

T. S. WHEELER.

X-Ray photography. F. SIMON (B.P. 276,678, 25.8.27. Ger., 25.8.26).—In X-ray photography, the difference in wave-length of the rays penetrating the object to be photographed is registered in visible colour tints by interposing between the object and the sensitive material a screen, the elements of which are formed of different transparent material of different extinction coefficient for X-rays, and at the same time rendered distinct by being coloured. A suitable screen is composed of starch grains of three kinds, of which the first are saturated with a lead salt, the second with a copper salt, and the third with an aluminium salt. Each of the kinds of starch is arbitrarily dyed with a different colour. W. CLARK.

Process of photogravure. E. STRAUB, Assr. to J. GRIFFITHS (U.S.P. 1,656,843, 24.1.28. Appl., 26.3.26. Fr., 10.2.26).—A plate is coated with a thin layer of dichromated gum arabic, dried, exposed through the object to be reproduced, rubbed with a solution of glycerin, water, and acetic acid until the image appears, and then washed in a solution of alcohol and acetic acid. A quick-drying varnish is then rubbed in to penetrate the bare parts of the plate, the plate is washed, dried, and inked with printing ink, after which the light-affected layer is removed with hydrochloric acid and the plate is etched. W. CLARK.

Compositions containing derivatives of carbohydrates [used as photographic supports]. H. J. HANDS, and SPICERS, LTD. (B.P. 282,980, 29.12.26).—Sheet material for forming photographic supports consists of compositions containing derivatives of carbohydrates having the empirical formula $(C_6H_{10}O_5)_n$, in particular the ethers and esters of cellulose and similar carbohydrates, in association with a relatively pure cellulose in a finely-divided form, and an opaque white pigment. The composition may be used for preparing compound sheets according to B.P. 281,803 (B., 1928, 187). W. CLARK.

Manufacture of bodies of desensitising action. R. SCHULOFF, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,653,314, 20.12.27. Appl., 10.12.26. Ger., 11.12.25).—See B.P. 262,816; B., 1928, 69.

Photometer (B.P. 275,597).—See XI.

XXII.—EXPLOSIVES; MATCHES.

Preparation of cellulose nitrate. BERL and BERKENFELD. **Decomposition of nitrocellulose.** RYAN and LANTZ.—See V.

PATENTS.

Initiating composition [for explosives]. E. VON HERZ (G.P. 443,551, 15.6.26).—A mixture of lead thiocyanate and lead azide is used. The mixture, which may be in the form of a mixed salt obtained by simultaneous precipitation, is more sensitive than lead azide alone. S. BINNING.

Composition producing yellow smoke. G. BOCK (G.P. 443,901, 5.2.26).—A mixture of powdered pitch and powdered sodium salts (*e.g.*, borax) is recommended. The volume of smoke may be further increased by addition of powdered glue. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Ferric salts as coagulants for activated sludge prior to filtration. F. W. MOHLMAN and J. R. PALMER (Eng. News-Rec., 1928, 100, 147—150).—Laboratory and large-scale experiments at Chicago indicate that ferric chloride is the most suitable reagent for conditioning activated sludge prior to filtration. The use of a mixture of ferric sulphate and ferric chloride, produced by chlorinating ferrous sulphate, is suggested as a means of overcoming difficulty in obtaining adequate supplies of ferric chloride at a reasonable cost.

C. JEPSON.

Dissolved oxygen absorption-time relation of activated sludge effluents. P. GAUNT and W. E. ABBOTT (J.S.C.I., 1928, 47, 14—16 T).—When diluted sewage or polluted water is incubated at 21°, the rate of dissolved oxygen absorption during the carbonaceous fermentation, lasting from 5 to 19 days, is represented by Phelps' equation, although the constant *K* has a more widely varying value than was previously recognised. With reasonably purified effluents, nitrification, commencing usually before the third day, causes rapid absorption of oxygen, and this equation does not then apply. A new equation is suggested to meet these conditions. The rate of acceleration of the absorption due to nitrification is shown, on certain simple assumptions, to be such as might be anticipated from the action of nitrite-producing organisms developing by binary fission. The time elapsing between successive fissions is shown to vary from one to three days. C. JEPSON.

Determination of dissolved gases in water. H. RICHTER (Chem.-Ztg., 1928, 52, 47).—Samples of water from pressure boilers should be cooled before releasing the pressure. Boiler water at 138° and 45 atm. had after releasing the pressure a temperature of 96° and showed no trace of dissolved oxygen. A similar sample, cooled to 30° before expansion, showed 0.2—0.3 mg. of oxygen per litre. C. HOLLINS.

PATENTS.

Purification and sterilisation of contaminated air by means of ozone. A. WOLFF (G.P. 445,502, 30.12.24).—The air mixed with ozone is brought in contact with aqueous solutions, atomised if necessary, containing acids or acid salts and catalytic material, *e.g.*, ferric chloride, until ozone is no longer present.

L. A. COLES.

Apparatus for automatically determining the amount of gas existing in water, and the automatic control of chemical operations. H. S. HATFIELD (B.P. 282,487, 20.9.26).—The apparatus refers particularly to the determination of temporary hardness in water. When a measured quantity of hard water is boiled the amount of carbon dioxide evolved by the decomposition of bicarbonates will be large in comparison with that merely dissolved in the water. The evolved gases are separated from the steam in a con-

denser, and then measured before and after passing over an absorbent of carbon dioxide. The measuring bells are connected to the outer wheels of a differential gear, and the inner wheel operates a pointer and/or electrical control arm by which means the hardness of the water may be indicated and/or caused to control the flow of a softening reagent. B. M. VENABLES.

Preventing the formation of incrustation or scale in boilers caused by salts of silicic acid. J. OSTERTAG (U.S.P. 1,657,443, 24.1.28. Appl., 17.6.27. Ger., 26.11.25).—The boiler is "blown off" and make-up water which has been treated so as to provide an excess of sodium carbonate over sodium silicate in the water in the boiler is added. The amount of blow-off and the amount and degree of alkalinity of the make-up water are calculated to maintain the total concentration below a predetermined maximum. M. E. NOTTAGE.

Prevention and removal of incrustation in boilers.

J. BILLWILLER (B.P. 281,361, 25.8.26. Addn. to B.P. 257,915; B., 1928, 72).—Fur and incrustation in boilers may be removed, or their formation prevented, by addition to the boiler or feed water of resin acids which have been slightly oxidised or polymerised by heating them above their m.p.; or resin acids containing "bound" water, obtained, *e.g.*, by hydrolysis of resins with alkali etc. and acidification, may be used. Gums may be added to the acids. B. FULLMAN.

Water-softening system. G. S. APELDORN (U.S.P. 1,646,270, 18.10.27. Appl., 26.6.25).—A zeolite water-softening plant comprising in particular a zeolite container connected at a relatively low point, by piping and valve, with the bottom of a second container of small capacity. The latter, which serves as a regenerating tank, is provided with means at the base for retaining solid reagent, and at the upper portion for supplying a stream of water to the reagent and for automatically cutting off the supply when the level of liquid in the vessel reaches a given height. When regeneration of the base-exchange media is necessary the zeolite container is drained, the required amount of salt is added to the second container, the two are placed in communication, and water is then supplied to the small vessel until the liquid reaches the desired level and the levels of liquid in the two containers are equalised.

W. T. LOCKETT.

Purification of water. W. H. GREEN and A. S. BEHRMAN, Assrs. to GEN. ZEOLITE CO. (U.S.P. 1,653,272, 20.12.27. Appl., 9.12.21).—The hardening constituents of water are precipitated by the addition of lime, freshly precipitated sludge, and aluminium sulphate.

H. ROYAL-DAWSON.

Purification of hard water by base-exchanging bodies. E. M. E. FRÉCHOU (U.S.P. 1,657,822, 31.1.28. Appl., 24.9.25. Fr., 19.12.24).—See B.P. 244,713; B., 1926, 390.

Apparatus for adding reagents to water. J. F. JONES (B.P. 282,193, 1.11.26).

Analysis of liquids (B.P. 282,168 and 282,170).—See I.

Base-exchange substance (U.S.P. 1,656,604).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MARCH 30, 1928.

I.—GENERAL; PLANT; MACHINERY.

Tube apparatus for crystallisation and evaporation. O. ZAHN (Chem. Fabr., 1928, 4—6).—The plant consists essentially of a slowly rotating iron tube, very slightly inclined to the horizontal, and provided with annular end pieces, by adjustment of which the height of the liquid passing through the tube is controlled. A current of air is drawn through in the direction opposite to that in which the liquid flows. The hot saturated solution is fed in at one end; the crystals formed are carried through with the mother-liquor and discharged continuously on to a filter or centrifugal. Brick or ebonite lining is specified for corrosive materials. When used as an evaporator, the tube is enclosed in suitable flues for heating. By connecting a heated tube with one through which cold air is drawn, evaporation and crystallisation may be effected continuously and automatically. S. I. LEVY.

Operating experiences with 1300 lb. steam pressure. J. ANDERSON (J. Inst. Fuel, 1928, 1, 131—160).—Eleven months' experience with a Stirling boiler operating at a pressure of 1300 lb. has shown that to avoid tube failures it is necessary to treat the feed water to prevent scale formation, although no difficulty arose when the same water was used in medium-pressure boilers. To prevent corrosion the oxygen content of the feed water must be reduced below 0.1 c.c./litre, and an appreciable caustic alkalinity must be maintained in the water. If, however, the concentration of alkali is too high, loss of turbine efficiency may be caused by deposition of solids on the blades. The use of trisodium phosphate in the boiler water in relatively small concentrations inhibits the formation of scale. The tensile strength of the metal of the tubes decreases with use, so that the usual method of testing when new gives too high a value for the strength after service. The general behaviour of the boiler at 1300 lb. was better than that at 300 lb. pressure, the water levels being stable and the moisture in the steam being low. A. B. MANNING.

Preheated air for boiler furnaces. P. H. N. ULANDER (J. Inst. Fuel, 1928, 1, 187—212).—The conditions are discussed under which the installation of an air preheater effects a useful economy of heat in boiler practice. The use of preheated air results in higher furnace temperatures and increased radiation, making possible a reduction in size of boiler and size of heating surface. Also a higher furnace rating can be maintained without serious loss due to incomplete combustion. Troubles such as "burning" of the links in a chain grate, uneven combustion, erosion of furnace walls, etc. may arise if preheated air is used in connexion with old type furnaces and grates, but with correctly designed

plant it is possible to operate safely with air temperatures of 250° or even higher. Examples of such installations are described and illustrated. A. B. MANNING.

Measurement of steam flow in works practice. H. C. ARMSTRONG and T. NORDENSON (J. Inst. Fuel, 1928, 1, 161—186).—Valuable economies can often be effected by control of the steam consumption in the various processes in a factory. A simple and inexpensive throttle orifice diaphragm meter for steam measurement is described and directions given for its use, together with diagrams for facilitating the necessary calculations. The theory of the instrument is developed in a second part of the paper [by M. BÄCKSTRÖM].

A. B. MANNING.

Measurement of surface temperature. D. F. OTTMER and H. B. COATS (Ind. Eng. Chem., 1928, 20, 124—128).—The study of heat transfer through a tube or other metal surface involves the measurement of the temperature of the surface. The use of thermo-couples is attended by difficulties, especially in avoiding heat conduction along the wire. The authors have obtained consistent results by plating a layer of nickel on the surface of a copper tube with nickel wires embedded in the plating. The hot junction has thus a broad interface and conducted heat is dissipated. The return wire of bright copper was simply wound round the unplated portion of copper tube, and all leads were insulated with rubber tubing. Details are given of tests with such copper-nickel thermo-couples with the tube surrounded by steam and having a current of cold water passing through it. Temperatures as much as 35° below that of the steam were read with a consistency of 0.05°. C. IRWIN.

PATENTS.

Furnace. W. G. WISE, ASST. to WISE FURNACE Co. (U.S.P. 1,658,364, 7.2.28. Appl., 19.11.27).—A burner for gaseous or vaporising liquid fuel is situated in a fire pot from the upper portion of which radiating ducts pass out to an annular hollow radiator drum which has (near its periphery) vertical gas ducts communicating with the ducts leading from the fire pot. At the top of the drum a number of radiating ducts lead the gases inwards towards the centre, whence they pass outwards again to the vertical ducts of another similar drum. Any number of such drums may be employed, and the gases finally pass out through a centrally disposed flue. C. O. HARVEY.

Coal-dust furnaces. SIEMENS-SCHUCKERTWERKE G.M.B.H. (B.P. 270,295, 25.4.27. Ger., 1.5.26).—The fuel is admitted axially downwards at the top and the primary air enters tangentially close by. Over the whole, or nearly all, of the interior surface of the

combustion chamber secondary air is admitted through numerous jets at such angles that the eddying, whirling motion is gradually decreased downwards so that the solid particles are kept away from the furnace walls by centripetal action.

B. M. VENABLES.

Regenerative furnaces. W. G. BEESTON (B.P. 282,841, 14.1.27).—A furnace chamber is connected through a valve chamber at each end to a pair of regenerator chambers, each valve being common to both regenerators and to the furnace; one valve communicates with an outlet stack, the other with an air-blast chamber. The air passes through one regenerator before the furnace, and the hot gases through the other regenerator after the furnace; on reversal the direction of the gases through the furnace does not reverse. [Stat. ref. to B.P. 11,402 of 1885.]

B. M. VENABLES.

Furnaces with chain or travelling grates. A. W. BENNIS (B.P. 283,243, 7.6.27).—A number of separate cells are situated under the chain grate and air of different humidity is supplied to them.

B. M. VENABLES.

Cooling towers. K. W. BRANCZIK (B.P. 282,932, 21.10.26).—A cooling tower is provided, near the mouth, with louver-like openings and curved deflector blades, which will convert a downwardly directed wind into an updraught within the tower, assisting the natural draught.

B. M. VENABLES.

Grinding mills. VICKERS, LTD., and L. D. PARKER (B.P. 282,989, 10.1.27).—A rotary cylinder has a number of V-shaped circumferential grooves formed on its inner surface, and balls run in the grooves, the balls being preferably considerably smaller than the grooves.

B. M. VENABLES.

Introduction of materials into high-pressure apparatus. SYNTHETIC AMMONIA & NITRATES, LTD., and H. A. HUMPHREY (B.P. 282,873, 27.9.26).—Two hydraulic rams enter a common work cylinder from opposite ends. The outer ends of the rams expand to a larger diameter forming double-acting pistons sliding in power cylinders. The feed material under low pressure is introduced through a port in the upper part of one end (say the left) of the work cylinder, and a port at the other end communicates with the high-pressure vessel. At the start the two rams are near each other at the left-hand end. The right-hand ram then moves to the right and draws in material. Both rams then move together to the right, closing the left port and opening the right port; the left ram usually moves rather faster than the right ram so as to compress the material. The left ram then continues alone and pushes the material into the high-pressure vessel. At completion both rams move together to the left for a new start, or they may be moved in inverse order to above to discharge the vessel.

B. M. VENABLES.

Apparatus for separating dust from suspension in gases and vapours. H. WARING, and ASSOCIATED LEAD MANUFACTURERS, LTD. (B.P. 282,849, 28.8.26).—A bag filter is constructed with tapering bag or bags supported at the upper smaller end by yielding means such as a counterweight which permits the bag to be shaken while in use. The dust is collected on the inside and the bags are preferably situated above a cyclone separator.

B. M. VENABLES.

Conditioning of gaseous fluids such as air. BRIT. ARCA REGULATORS, LTD., and T. LINDSAY (B.P. 282,915, 9.10.26).—In an apparatus where gases are conditioned by saturating with fluid at a lower temperature and are then re-heated to the required dry-bulb temperature, the variations in temperature of the saturating liquid are caused to alter an aperture through which a non-compressible fluid is pumped at a constant rate. By this means the pressure of the non-compressible fluid will vary according to the aperture, and the variations of pressure will operate a servo-motor which controls the supply of heat or cold. A similar device is used to control the re-heating of the gas after saturation.

B. M. VENABLES.

Apparatus for bringing liquids and gas or vapours or both gas and vapours into intimate contact, and liquid-distributing devices adapted for use therein. W. C. HOLMES & CO., LTD., C. COOPER, and D. M. HENSHAW (B.P. 283,028, 13.4.27).—In gas washers of the "brush" or other rotating type, buckets attached to the rotor are employed to lift and distribute the liquid over the effective surface without immersing much of the rotor and making use of only a small bulk of liquid. The buckets are specially shaped so as to discharge liquid at a rate proportional to the vertical depth of the effective surface below any point of discharge. The buckets are usually in pairs, one bucket of each discharging while rising 90° from horizontal to vertical and the other bucket while dropping from vertical to horizontal.

B. M. VENABLES.

Purifying separator for liquids. J. SCHAEFER (B.P. 272,927, 15.6.27. Fr., 15.6.26).—A settling chamber for separating, e.g., water from petrol, is provided with a float valve and automatic siphon so that the heavier liquid is automatically drained off at intervals.

B. M. VENABLES.

Filters and strainers for liquids. D. A. COATES (B.P. 282,846, 29.4.27).—For a filter of the type in which a helical or conical coiled spring is used as the filtering medium, several methods of expanding or contracting the spaces between the coils are described.

B. M. VENABLES.

Degasification of liquids. SOC. ITAL. PIRELLI (B.P. 276,987, 25.8.27. Italy, 31.8.26).—The liquid is supplied to near the centre of the uppermost of several rotating saucers or shallow cones, which alternate with other fixed collecting cones with inturned edges; the latter return the liquid to the centre of the next rotating saucer below and so on. While under treatment the liquid is subjected to a vacuum, and if necessary to heat, and it is stored in vessels either under vacuum or which have expansible walls and are completely filled.

B. M. VENABLES.

Apparatus for purifying and cooling gases. A. L. MOND. From METALLBANK & METALLURGISCHE GES. A.-G. (B.P. 282,939, 3.11.26).—The gas is caused to pass through a number of convergent and divergent passages with sharp changes of direction at the narrowest parts. The passages may be mounted on a drum or other rotary support which dips into liquid, and the depth of submergence in the liquid may be used to vary the available area of the passages according to the amount of gas under treatment.

B. M. VENABLES.

Grinding mill. J. B. SEDBERRY (U.S.P. 1,659,499, 14.2.28. Appl., 4.4.24).—See B.P. 231,386; B., 1925, 385.

Centrifugal treatment of liquids. K. J. SVENSSON and K. A. P. NORLING (U.S.P. 1,659,222, 14.2.28. Appl., 14.12.26. Swed., 11.1.26).—See B.P. 264,130; B., 1927, 287.

Superheater boilers. T. E. ROBERTSON. From POWER SPECIALTY Co. (B.P. 283,031, 20.4.27).

Joint-making packing for high-pressure vessels etc. A.-G. BROWN, BOVERI, & Cie. (B.P. 264,524, 14.1.27. Ger., 16.1.26).

Hollow grinding bodies for ball-, drum-, and tubular mills. O. KORDT (B.P. 284,580, 12.12.27).

Drying of viscid liquids (B.P. 282,480).—See V. Catalysts (B.P. 281,218).—See X.

II.—FUEL; GAS; TAR; MINERAL OILS.

Differential index of the coking power of coal. A. W. COFFMAN and T. E. LAYNG (Ind. Eng. Chem., 1928, 20, 165—170; cf. Layng and Hathorne, B., 1925, 195).—By use of a softening and solidification point test a graphical means of determining an index number called the agglutinating index for any coal may be arrived at. An initial softening temperature for each coal is indicated by an increase of pressure which assumes a maximum value and then rapidly decreases to normal. Study of that portion of the temperature-pressure curve falling between the initial softening point and the point of maximum pressure gives a true measure of the range of plasticity of a coal. The values d_p/d_i calculated at regular intervals and plotted against the time in minutes show an increase to a maximum value which is taken as the differential index or agglutinating index of a coal. To be comparable all data must be obtained at the same rate of heating. From the appearance of the curves under such test conditions, for a coal to coke the differential index must be above 20 and the total energy must be used in a comparatively short time giving rise to a peak in the curve. These two conditions may be fulfilled in the case of the more poorly coking coals by varying four factors which govern the agglutinating value, viz., the rate of heating at which the coal is carbonised, the previous conditioning of the coal, the amount of weathering to which the coal has been subjected, and the chemical interaction taking place between the coal constituents.

H. S. GARLICK.

Low-temperature coke from coking coals. M. DOLCH (Oesterr. Chem. Ztg., 1928, 31, 11—15).—An essential condition for the economic development of low-temperature carbonisation is that the income derived from the coke produced should cover the cost of the raw coal. Only the use of coking coals or mixtures containing coking coals can fulfil this condition, but even so the value of the coke will depend largely on the choice of a suitable type of carbonisation plant. In evaluating low-temperature cokes the calorific value of the volatile matter evolved on heating to 1150° is of more importance than its actual amount (cf. B., 1926, 651).

A. B. MANNING.

Use of the bomb for the determination of carbon and hydrogen [in combustible material]. J. W.

WHITAKER (Fuel, 1928, 7, 63—65).—Carbon and hydrogen are determined by a modification of Goutal's method (B., 1923, 1162 A). After combustion in the bomb, in the bottom of which is placed a crucible containing anhydrous calcium chloride, the gases are gradually discharged through (1) a weighed U-tube containing pumice soaked in sulphuric acid, (2) a gas sampling tube, (3) a wash-bottle containing water saturated with carbon dioxide, and (4) a meter. The carbon content of the fuel is then calculated from the total volume of gas in the bomb and the percentage of carbon dioxide therein. The hydrogen is determined from the increase in weight of the crucible containing calcium chloride and the U-tube through which the gas is passed. The presence of sulphur does not seriously interfere with the hydrogen determination.

A. B. MANNING.

Unsaturated hydrocarbons in the gases from the carbonisation of coal. A. B. MANNING, J. G. KING, and F. S. SINNATT (Dept. Sci. Ind. Res., Fuel Res. Tech. Paper No. 19, 1928, 19 pp.).—Ethylene, propylene, and butylene are determined in a coal gas by passing a known volume (about 10 cub. ft.) of the gas through bromine, after a preliminary oil scrubbing to remove the vapours of volatile liquids. The bromine compounds of the unsaturated gases are distilled under reduced pressure and the olefines are regenerated by the action of a zinc-copper couple on a weighed quantity (about 2 g.) of the distillate in solution in 95% alcohol. The olefines in the gas so obtained are determined by (1) absorption of the propylene and butylene in 87% sulphuric acid, making a correction for the small concurrent ethylene absorption, followed by absorption of the ethylene in fuming sulphuric acid, (2) combustion of a second portion of the gas over copper oxide and measurement of the carbon dioxide produced. It is then possible to calculate the ethylene, propylene, and butylene content of the original gas. By extraction of the residue from the distillation of the bromine compounds with alcohol the butadiene tetrabromide present can be isolated and thence an approximate value of the butadiene content of the gas obtained. A typical low-temperature coal gas contained ethylene 1.18%, propylene 1.16%, butylene 0.68%, butadiene 0.15%; a high-temperature, horizontal-retort gas contained ethylene 1.90%, propylene 0.28%, butylene 0.08%, butadiene 0.02%. Results for other types of gases, including a series produced by the carbonisation of coal at temperatures from 400° to 700° (cf. Sinnatt, King, and Linnell, J.S.C.I., 1926, 45, 385 r), are given and briefly discussed.

A. B. MANNING.

Gas analysis. H. M. STANLEY and A. W. NASH (Gas J., 1928, 181, 391).—Determination of paraffin hydrocarbons in gases of unknown composition is often difficult by the explosion method, and a modified procedure of oxidation by copper oxide at 900° is available. Although this oxidation takes place somewhat slowly, and generally produces less carbon dioxide than an explosion, it is considered preferable, especially with gases rich in nitrogen.

R. H. GRIFFITH.

Method of fractionating natural gasoline. D. B. KEYES (Chem. Met. Eng., 1928, 35, 92).—The preheated oil is passed into a stripping column (steam being used

as the heating medium throughout) from the bottom of which pure absorbing oil and water are withdrawn; the oil after being cooled is separated from the water and returned to the system. The vapours from this column, consisting of all the gases, all of the product, and a little of the absorbing oil, pass to the first condenser, which is run warm. Part of this condensate is returned as reflux and part sent on to the next fractionation column. The second condenser is a cold or total condenser. The condensate from this enters a third fractionating column. The product is cooled, separated, and collected in a storage tank, whilst the uncondensed gases are sent to the compressors, coolers, and water-separators along with the uncondensed gas from the condensers on the stripping column. A second absorption in an absorbing column then takes place, an absorbing oil of lower b.p. being used. The gas-oil solution is stripped in a stripping column, and the pure absorbing oil returned to the absorber. The crude product after refining in a fractionating column passes on to the storage tank, the product in which is again fractionated to give the final pure product.

H. S. GARLICK.

Cracking experiments by the Dubbs process with distillates from Hanoverian petroleum. R. HEINZE (*Petroleum*, 1928, **24**, 237—241).—The yield and nature of cracked spirit obtained with Hanoverian petroleum are compared with those given by American oils. A gas-oil fraction, b.p. about 200—360°, was cracked under 14 kg./cm.² pressure, and gave 36% of motor spirit, 20% of gas oil, and 23% of fuel oil, whilst under 19 kg./cm.² the yield of gas oil decreased to 16%, and under 21 kg. pressure was still less, but no regularities appear in the proportions of the other fractions. The motor spirit produced under 21 kg. pressure contains 23% of aromatic, 27% of naphthene, and 40% of paraffin hydrocarbons, and has pronounced anti-knock properties. A heavier petroleum fraction, b.p. about 240—400°, was also cracked under 5.25 kg. to two different end-points, and gave a yield of about 30% by wt. of motor spirit, with a high aromatic and low naphthene content, and with a Ricardo compression ratio of 5.8. Refining of the cracked distillates was in all cases carried out with plumbite solution and with sulphuric acid, but the quantities necessary varied with the different products.

R. H. GRIFFITH.

Steam-temperature control in distillation practice and in paraffin manufacture. E. BELANI (*Petroleum*, 1928, **24**, 199—201).—When steam is used in distillation of oils and tars, or in refining of paraffin wax, control of its temperature and pressure is very important; if the steam is raised by a Benson boiler or similar apparatus in which water is heated above its critical temperature and pressure, and is converted into steam without boiling, it will be too hot for such purposes. Any device for lowering the temperature of the steam must provide a steady exit temperature and pressure regardless of inlet variations, and a machine for effecting this has now been perfected. Steam enters at the bottom of a vertical vessel and meets a fine spray of water; this is completely vaporised, and the exit steam will have a temperature depending on the quantity of water used. Thermometers at the inlet and outlet operate a relay which opens or closes a valve controlling the amount

of this added water, so that fluctuations at the inlet are immediately compensated.

R. H. GRIFFITH.

Preheated air for boiler furnaces. ULANDER.—See I. **Synthetic phenol.** HALE and BRITTON.—See III.

PATENTS.

By-product coke oven. J. V. K. BORLAND (U.S.P. 1,658,796, 14.2.28. Appl., 13.9.23).—To obtain a uniform distribution of fuel gas from a main supplying a battery of coke ovens, the gases are maintained at a substantially constant temperature throughout the entire length of the main.

A. B. MANNING.

Cleaning of coal. C. R. DOWNS and C. H. STRANGE, Assrs. to HIRSCH-LEDERER SYND., INC. (U.S.P. 1,656,270—1, 17.1.28. Appl., 8.5.25).—(A) Coal and its impurities are separated by gravity in a medium formed by a suspension of a finely-divided solid in a liquid, the sp. gr. of the medium being intermediate between that of the coal and that of the impurities. The finely-divided solid separated from the washed coal by screening is cleaned from fine coal and other impurities by flotation and returned to the gravity separator. In (B) the finely-divided solid used for the suspension is made up largely of finely-divided material separated from the coal itself.

A. B. MANNING.

Washing of coal, ores, and like substances. L. HOVOIS (B.P. 266,723, 22.2.27. Belg., 25.2.26. Addn. to B.P. 258,753; B., 1926, 971).—The principles of the main patent and the additional patent B.P. 265,202 (B., 1928, 114) are applied for the further separation of the products. By the use of a projecting box with several adjustable openings and an adjustable current of water the material is re-graded and subdivided; hoppers and chutes are arranged to receive the various products obtained.

A. C. MONKHOUSE.

Elaborating the products arising during the liquefaction of coal by hydrogenation. DEUTS. BERGIN-A.-G. F. KOHLE U. ERDÖLCHEMIE (B.P. 262,738, 5.11.26. Ger., 11.12.25).—A small quantity, e.g., 20%, of liquid aliphatic hydrocarbons such as gas oil is added to the products of hydrogenation which have been previously freed from water and constituents boiling below 280°. Asphalt and solid insoluble substances are precipitated and the oil is separated. On keeping, a further quantity of soft asphalt is precipitated; the precipitating medium is recovered from the fractions by steam-distillation.

A. C. MONKHOUSE.

Distillation of coal. B. ZWILLINGER, Assr. to TAR & PETROLEUM PROCESS Co. (U.S.P. 1,656,617, 17.1.28. Appl., 14.7.22).—A thin horizontal layer of coal is distilled in an oven chamber heated from beneath by a number of longitudinal, parallel flames which are independently regulable, and the distillates so produced are collected.

A. B. MANNING.

Distillation of carbonaceous material. L. C. KARRICK (B.P. 261,362, 2.11.26. U.S., 16.11.25).—A highly reactive fuel is obtained by distilling coal etc. in continuous vertical retorts internally heated by superheated steam or gas. Waste steam is admitted to superheaters situated in the lower half of the setting between the retorts and is heated at 540—940° by

the burning of fuel gas and air, both preheated in the upper portion of the setting. The steam or gas is admitted at the mid point of the retorts, and additional steam or gas is admitted at the bottom for cooling purposes.

A. C. MONKHOUSE.

Coking of coal. URBANA COKE CORP., Assees. of S. W. PARR and T. E. LAYNG (B.P. 263,785, 13.12.26. U.S., 2.1.26; cf. Parr, B., 1926, 697).—Coal is fed from a hopper into an inclined rotating drum which is heated by waste flue gases as well as by supplementary, automatically controlled gas burners. It is thereby preheated, out of contact with air, nearly but not quite to the pasty stage. In order to economise heat as far as possible the flue gases after leaving the preheater pass through flues in the hopper itself before being discharged to air. The coal is transferred directly from the preheater into vertical retorts, which are already heated to the desired temperature (750°), and is therein carbonised. The retorts are of metal, circular in cross-section and slightly tapered. They are heated principally by radiation from the walls of the heating chamber in which are flues provided with gas burners. After circulating in the flues the hot gases pass up around the retorts and thence to the preheater or to a recuperator for heating the air supplied for combustion. When the coal is completely coked it is discharged into a storage chamber in which it is cooled; air passages surround this chamber so that the sensible heat of the coke may be utilised in pre-heating the air required for combustion.

A. B. MANNING.

Treatment of carbonaceous material. R. V. FARNHAM (B.P. 283,717, 18.12.26).—An apparatus for drying or carbonising fuels or other materials consists of a number of elongated rectangular sections bolted together one above the other. A "retarder," consisting of a tray of channel or H-section which can be rotated about a horizontal axis, is arranged in each section in such a manner that by its rotation the material is delivered from that section to the one below, and by suitably adjusting the periods of rotation the material can be subjected to the heating medium for any desired period. The material is fed into the top section by means of arms rotating in a cylindrical hopper, and may be withdrawn similarly at the bottom. Means are provided for passing heated gases through the apparatus.

A. B. MANNING.

Apparatus for the manufacture of briquettes. S. R. ILLINGWORTH, and ILLINGWORTH CARBONIZATION CO., LTD. (B.P. 284,007, 19.10.26. Addn. to B.P. 258,327; B., 1926, 1003).—The main patent is modified in that the moulds on the outside of the rotating drum are detachable, being secured in position by endless links engaging the walls of the moulds. The moulds may consist of trays divided into a number of compartments, each of which forms a mould, the links then engaging the outside walls of the end compartments of each tray. The links have teeth on their outer surfaces, which mesh with teeth on the rollers supporting the drum, and by which the latter is driven. The coal, after being fed into the moulds, may be compressed by a ram working in a cylinder carried in the casing wall.

A. B. MANNING.

Formation of reconstructed carbonaceous fuel. W. B. RULON (U.S.P. 1,656,364, 17.1.28. Appl., 28.7.25).—Finely-divided solid carbonaceous material is agitated with a liquid towards which it is inert, and a volatile liquid which has an affinity for the solid is then added. The plastic "amalgam" produced in the form of small pieces is compressed into relatively large lumps, which are heated to render them solid and to reduce the percentage of volatile matter therein.

A. B. MANNING.

Utilising powdered fuel. Utilising pulverulent or powdered carbonaceous material. J. J. C. BRAND and B. LAING (B.P. 281,098 and 281,588, [A, B] 26.5.26).—(A) Powdered fuel of size such that 85% is below 200-mesh is fed by worm conveyor into a vessel and is kept in agitation by the admission of air or inert gas at the bottom, so that the normal inter-air space is increased by 30–60%. Air at 5–100 lb./in.² is used to force the powdered fuel to the burners, where the supply is controlled by an inverted needle valve. Baffles impart a whirling motion to the fuel-air mixture, and gauze diaphragms are used to avoid flash-backs from the burner. A portable form of apparatus is described. (B) For the burning of powdered fuel in existing marine etc. boiler furnaces a preliminary combustion chamber lined with firebrick is used. The chamber has an inclined front wall with suitable burners pointing vertically downwards. The fuel and primary air are met at the front wall by the secondary air, which diverts the burning fuel to the rear wall of the chamber over the fire-bridge into the furnace. The temperature is maintained below that of slag formation by cooling externally with air or water.

A. C. MONKHOUSE.

Producing or reactivating active carbon. VER. F. CHEM. IND. A.-G., Assees. of E. KÜCHLER (B.P. 260,567 and Addn. B.P. 265,916, 11.10.26. Ger., [A] 27.10.25, [B] 15.2.26).—(A) Carbon is heated in a container with porous or permeable walls, outside which is circulated a gas containing oxygen, the material being activated by the action of the gas which diffuses through the walls. At the same time a current of the gas is passed directly into the container, in quantity, however, insufficient in itself for activation. (B) In a modification of the above process the container is made of narrow dimensions or is provided with internal channels for the passage of the gases in order to accelerate the activation. The container may be heated by furnace gases, and, if desired, the activating gases may be preheated.

A. B. MANNING.

Manufacture of producer gas. W. M. CROSS (U.S.P. 1,657,371, 24.1.28. Appl., 28.4.27).—Producer gas is obtained by passing a mixture of air and steam (automatically maintained at a temperature above the predetermined temperature of reaction) along with pulverised carbonaceous material to a reaction zone.

C. O. HARVEY.

Roasting of used or waste gas-purifying substances. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (B.P. 271,854, 12.5.27. Ger., 25.5.26).—Waste gas-purifying masses containing sulphur are roasted in a furnace through which they pass in the same direction as the roasting gases. The oxidation of the volatile organic compounds and ammonia is thereby practically

completed, and the resulting gases can be used directly in the lead chamber process without unduly increasing the consumption of nitric acid. A further improvement is effected by preheating the air used for roasting, *e.g.*, by passage over the hot roasted mass. A. B. MANNING.

Catalytic process for desulphurising hot gases. H. FLEISSNER (Aust. P. 106,443, 18.6.25).—A mixture of the hot gas with air is passed over a catalyst obtained by roasting spathic iron ore or other minerals containing ferrous carbonate. L. A. COLES.

Hot desulphurisation of gases derived from the distillation or pyrogeneration of solid, liquid, or other combustibles. SOC. INTERNAT. DES PROC. PRUD'HOMME HOUDRY (B.P. 267,138, 4.3.27. Fr., 4.3.26).—The gases are passed at 250° through a purifier containing a metal or metallic oxide, *e.g.*, nickel oxide, to remove hydrogen sulphide, a purifier containing oxide of iron or copper to remove organic sulphur, and, if the gases contain water vapour, a third purifier similar to the first to remove hydrogen sulphide evolved from the second purifier. Regeneration is effected by air or by water-gas mixed with steam and heated at about 350°.

A. C. MONKHOUSE.

Low-temperature distillation of coal. INTERNAT. COMBUSTION ENGINEERING CORP., Assecs. of W. RUNGE (B.P. 267,095, 2.2.27. U.S., 4.3.26; cf. B.P. 242,623; B., 1927, 162).—Finely-powdered coal passes down a vertical retort counter-current to an ascending stream of hot gases, and is thereby carbonised to give a coke consisting substantially of hollow particles filled with combustible gas. The coke is discharged into a conduit through which it is conveyed by a current of cool combustible gas, the latter thereby taking up part of the heat of the coke. After separation of the coke, the gas is passed to a specially designed preheater in which part of it is burnt and mixed with the remainder, and with other gases if required, in such proportion as to bring their temperature to that necessary for the distillation process. The mixed gases thence pass to the retort.

A. B. MANNING.

Continuous distillation of tree branches, vines, etc. E. A. BARDET and R. MALBAY (F.P. 621,350, 6.1.26).—The wood, cut up into small pieces, is carbonised in a continuous vertical retort, and the gases formed are separated into two portions, one containing the water evolved during the first stage of the heating, and the other suitable for use, *e.g.*, for driving agricultural machinery.

L. A. COLES.

Distillation of pyroligneous acid. SOC. DES PROD. CHIM. DE CLAMECY (B.P. 269,927, 22.4.27. Fr., 22.4.26).—Crude pyroligneous acid is distilled and the vapours evolved during the first stage of the distillation are led into a column in which they come into contact with benzene or a similar liquid with which methyl alcohol forms a binary mixture of minimum b.p. The vapours from this column are condensed, the methyl alcohol, together with some acetone and methyl acetate, extracted with water, and the benzene is returned to treat a further quantity of the pyroligneous acid vapours. The vapours evolved during the next stage of the distillation are led to a second column in which, by suitable adjustment of the reflux, the bulk of the acetic acid is condensed in a

concentrated form, water, allyl alcohol, and oily impurities passing to the receiver. These oily impurities are further treated with water to recover allyl alcohol.

A. B. MANNING.

Resolution of aqueous emulsions of tar. B. JOHNSON (B.P. 284,401, 27.10.26).—Tar emulsions are resolved by the addition of a small quantity of a carboxylic acid, *e.g.*, resin acid, oleic acid, stearic acid, etc., which is soluble in the tar, but insoluble in the water, the mixture being then maintained at 60–70° until separation is complete. When treating tars which contain no ammonia it is convenient to add a small quantity of an alkali so that the separated water has an alkaline reaction.

A. B. MANNING.

Recovery of sulphuric acid from the acid tar of benzol purification. C. STILL (B.P. 277,619 and Addn. B.P. 281,547, [A] 16.5.27, [B] 31.5.27. Ger., [A] 20.9.26, [B] 22.12.26).—(A) The acid tar is mixed with concentrated ammonium sulphate liquor from the saturator, and is maintained at 55° by means of a steam coil. The acid resinous constituents rise to the surface on being kept, and after separation and removal of the ammonium sulphate liquor are heated to remove benzol hydrocarbons. Tar or tar oils are added to the product, and the mixture is washed with water before discharge. (B) The operations take place at moderate temperature without the application of external heat.

A. C. MONKHOUSE.

Manufacture of water-soluble humic acid derivatives. O. L. BEER (G.P. 443,339, 29.3.23).—Humic acid or one of its alkali salts is treated with sulphur dioxide or sulphites at ordinary or increased pressure in presence or absence of aldehydes, and, if desired, is treated with chlorine. *E.g.*, an alkaline extract of brown coal is boiled with 38% sodium hydrogen sulphite solution; the resulting brown solution has tanning properties, gives a soluble residue on evaporation, and forms insoluble heavy-metal salts. Tanning materials are also obtained by heating alkali humate with sodium sulphite and formaldehyde solutions, or by the action of chlorine on alkali humate solution. Raw humus (peat etc.) may be warmed with sodium carbonate solution, sulphite added, and heating continued under pressure; sulphur dioxide then precipitates a sulphited humic acid soluble in water.

C. HOLLINS.

Manufacture of a bituminous composition. W. P. BENTLEY (U.S.P. 1,659,554, 14.2.28. Appl., 18.4.24).—A suitably graded non-bituminous mineral is mixed with a crushed bitumen-containing rock, and there is added to the mixture first a volatile liquid capable of liquefying the bitumen in the rock, and then such a quantity of a bituminous flux in liquid condition that the total quantity of bitumen is just sufficient to coat the mineral and fill the voids in the mixture.

A. B. MANNING.

Ammonia-saturator apparatus. J. BECKER, Assr. to KOPPERS Co. (U.S.P. 1,654,159, 27.12.27. Appl., 11.2.21).—An apparatus for use in the application of the process described in U.S.P. 1,375,483 (B., 1921, 426 A) comprises a tank supplied with sulphuric acid and gas containing ammonia, and fitted with a rotor which promotes intimate mixture and eliminates back-

pressure. The precipitated salt falls to the bottom of the tank, from which it is removed by a compressed air ejector.

T. S. WHEELER.

Cracking of petroleum oil. R. T. POLLOCK, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,658,415, 7.2.28. Appl., 20.12.20).—The oil is cracked under pressure by passage through a heating coil and then enters a vapour chamber whence the vapours pass to a series of dephlegmating columns, the condensates from which are passed, under applied pressure, to independent heating zones, maintained at temperatures above that of the original heating coil.

C. O. HARVEY.

Cracking of [hydrocarbon] oils. J. C. CLANCY (U.S.P. 1,658,116, 7.2.28. Appl., 2.9.22).—Gasoline-like products are produced from heavier hydrocarbon oils by passing them (in the presence of hydrogen) in admixture with molten material containing an alkali cyanide through a retort packed with a metallic catalyst (nickel) maintained at a cracking temperature. The molten material is supplied to, and the mixture of this material with unvaporised oil is withdrawn from, the retort through liquid-sealed passages. The evolved vapours are collected.

C. O. HARVEY.

Treatment of natural-gas, casing-head, and natural gasolines etc. II. B. BERNARD, Assr. to SINCLAIR OIL & GAS Co. (U.S.P. 1,658,322, 7.2.28. Appl., 22.7.24).—The crude gasoline product flows in a helical path down a tower in counter-current to vaporised constituents (which are flowing upwardly), no mixing of vapours and liquid being attempted (*i.e.*, by spraying or bubbling). The vapours and gases are withdrawn at the top and the treated gasoline product at the base of the tower.

C. O. HARVEY.

Manufacture of valuable liquid and other hydrocarbons and derivatives thereof by the destructive hydrogenation of mineral coal and other solid carbonaceous materials. I. G. FARBENIND. A.-G. (B.P. 256,964, 10.8.26. Ger., 14.8.25).—In the catalytic hydrogenation of coal and other carbonaceous materials (other than lignite or peat) at elevated temperatures and under pressures of at least 50 atm., an increased yield of liquid products is obtained and choking of the apparatus by coke-like products is avoided by mixing the initial material with lignite or peat or both, whereby a material is obtained which, under high pressures, may be caused to flow like a viscous liquid.

C. O. HARVEY.

Manufacture of valuable liquid and other hydrocarbons and derivatives thereof by the destructive hydrogenation of carbonaceous materials. I. G. FARBENIND. A.-G. (B.P. 257,256, 10.8.26. Ger., 20.8.25).—Motor fuel of a non-detonating character, lubricating oils, etc. are produced from carbonaceous materials (coal, wood, bone tar, mineral oil residues, bitumen, resins, etc.) by interaction at high temperatures and pressures with water vapour or with gases containing hydrogen and oxides of carbon, in the presence of catalysts capable of forming methyl alcohol by the catalytic hydrogenation of oxides of carbon (vanadic acid, manganese, uranium, zinc, and titanite oxides, etc.). The initial material may be submitted to a preliminary hydrogenation, and it is preferable to allow only gases

or finely-divided material to come into contact with the catalyst (in a separate chamber).

C. O. HARVEY.

Distillation of oils from oil shale or coal or similar material. R. H. CROZIER (B.P. 283,639, 13.10.26).—The distillation of oil from shales and coals of an intumescent character is facilitated and the yield is improved by carrying out the operation in a continuous vertical retort heated by gas burners, the upper portion of the retort, into which the raw material is introduced, being the hottest portion (about 400°). By this arrangement the pitch-like constituents are rapidly vaporised and a semi-coke is produced which readily flows downwards into the cooler zones of the retort, where it is further distilled.

C. O. HARVEY.

Refining of oil. E. C. R. MARKS. From SIMPLEX REFINING Co. (B.P. 283,383, 20.4.27).—In the treatment of oils under reduced pressure, loss of vacuum due to leakage through mechanical pumps is avoided by causing the oil to circulate automatically without mechanical aid. *E.g.*, in the distillation of lubricating oil, the vapours pass upwards through a dephlegmating column down which the condensate is caused to flow. The condensate collects in a chamber at the base of the column which is connected with a U-shaped pipe which passes downwards for some distance and then upwardly, finally discharging into the top of the dephlegmating column. The condensate collects in the U-tube and a hydrostatic head is established. By introducing a vapour (*e.g.*, steam) into the longer arm of the tube, a lighter mixture is produced, and the oil is thereby caused to rise and to discharge itself into the dephlegmator.

C. O. HARVEY.

Refining of oil. J. C. BLACK (U.S.P. 1,656,997, 24.1.28. Appl., 5.4.20).—The oil is treated at a reactive temperature with acid-treated clay.

C. O. HARVEY.

Refining of [hydrocarbon] oils. A. SCHWARZ, Assr. to PETROLEUM SAND PRODUCTS CORP. (U.S.P. 1,656,990, 24.1.28. Appl., 14.6.24. Renewed 3.12.27).—Hydrocarbon oils containing desirable and undesirable unsaturated hydrocarbons are freed from the latter by treatment with a suitable mixture of sulphuric acid and saturated hydrocarbons.

C. O. HARVEY.

Refining of petroleum oils. P. McMICHAEL, Assr. to HYDROCARBON REFINING PROCESS Co. INC. (U.S.P. 1,658,171, 7.2.28. Appl., 17.11.24).—Cracked petroleum oils are treated in a series of washing chambers successively with a solution of caustic soda (with or without the addition of sodium sulphide), an alkaline sodium chromate solution, water, a 70–83% solution of sulphuric acid, water, and finally with a solution of caustic soda.

C. O. HARVEY.

Removal of sulphur and sulphur compounds from hydrocarbon oil. G. L. WENDT, Assr. to STANDARD OIL Co. (U.S.P. 1,658,505, 7.2.28. Appl., 16.7.24).—The oil is treated with an alcoholic solution of sodium plumbite.

C. O. HARVEY.

Purification of crude benzol. I. G. FARBENIND. A.-G. (B.P. 258,576, 9.9.26. Ger., 15.9.25).—Crude benzol is substantially freed from sulphur compounds and converted into a mixture suitable for distillation for the production of pure benzol and other low-boiling hydrocarbons by treatment with hydrogen (or gases

containing it) under pressure (150—200 atm.) at temperatures of about 450° and in the presence of suitable catalysts (mixtures of molybdic acid with magnesium or zinc oxide etc.). Under these conditions the benzol itself is practically unaffected, and the emulsion losses inherent in the acid-refining process are avoided.

C. O. HARVEY.

Mineral oil compositions for lubricating, insulating, and other purposes. R. T. VANDERBILT CO. INC., Assees. of A. A. SOMERVILLE (B.P. 269,840, 9.10.26. U.S., 20.4.26).—Lubricating oils (particularly those for use at high temperatures or in air compressors) and insulating oils are rendered more stable and less liable to undergo atmospheric oxidation by the addition of small amounts (0.5 to a few per cent.) of condensation products of aldehydes with nitrogenous bases (e.g., the condensation product obtained by the interaction of acetaldehyde and aniline).

C. O. HARVEY.

Production of clear [lubricating] oils soluble in water. A. HORN (B.P. 281,476, 19.7.27).—A vegetable oil, such as castor oil, is heated to about 300°, allowed to cool, and then heated to 100—110° with hydrochloric acid vapour with the addition of a contact substance, e.g., calcined aluminium chloride. After cooling, oxygen, air, or ozone is blown through the oil at 150—200° in an open vessel to remove the acid. A contact substance is added such as hydroxides, carbonates, percarbonates, or perborates of the alkalis, stannous chloride, or bases such as resorcinol, pyridine, quinone, quinol, quinoline, or hydroxylamine and trimethylamine in hydrochloric acid or alone. Treatment is continued at 200° and lasts 4—5 hrs.

A. C. MONKHOUSE.

Manufacture of motor fuels. I. G. FARBENIND. A.-G. (B.P. 259,944, 2.10.26. Ger., 19.10.25).—Fuels for use in internal-combustion engines are improved in regard to absence of detonation, more suitable boiling range, ease of starting the engine, etc. by the addition of suitable proportions of products obtained by the destructive hydrogenation of coal, tars, oils, or other carbonaceous materials.

C. O. HARVEY.

Fuel for internal-combustion engines. S. ISERMAN, W. VERNET, and E. Q. MOSES, Assrs. to BOYCE & VEEDER CO., INC. (U.S.P. 1,654,259, 27.12.27. Appl., 22.7.24).—*o*-Nitrotoluene (0.1%) is added to petrol as a decarbonising agent. The use of 1:4-ditoluidino-anthraquinone (0.001%) to give a distinctive greenish-blue colour to petrol so treated is also claimed.

T. S. WHEELER.

Reactivation of adsorptive material. W. S. BAYLIS, Assr. to FILTROL CO. (U.S.P. 1,654,629, 3.1.28. Appl., 5.4.26).—Adsorptive material used to treat oil is removed in a filter-press in which the filter-medium is formed of a metallic fabric, and is then treated *in situ* with a mixture of superheated steam and air at 400°.

T. S. WHEELER.

Manufacture of gasoline. O. BEHMER, Assr. to TEXAS CO. (Re-issue 16,877, 14.2.28, of U.S.P. 1,585,496, 18.5.26).—See B., 1926, 623.

Recovery of acid and resinous matter from acid sludges formed in the refining of mineral oils.

W. DEMANN (B.P. 258,241, 25.8.26. Ger., 9.9.25).—See G.P. 436,242; B., 1927, 436.

Sulphonic acids from tar oils (G.P. 445,645). **Cresols etc. from creosote liquors** (G.P. 444,971).—See III. **Absolute alcohol** (B.P. 283,701).—See XVIII.

III.—ORGANIC INTERMEDIATES.

Development of synthetic phenol from benzene halides. W. J. HALE and E. C. BRITTON (Ind. Eng. Chem., 1928, 20, 114—124).—The hydrolysis of halogenobenzenes with caustic soda solution at high pressures and temperatures up to 370° was studied by means of a rotating bomb of heavy piping. The optimum concentration of sodium hydroxide was concluded to be 8%. Higher strengths give an undue formation of tar, whilst lower strengths increase the production of diphenyl ether in place of phenol. The reaction is found to be unimolecular, and the concentration of alkali has no effect on the velocity of hydrolysis. The effect of catalysts is not great except at lower temperatures; metallic copper and cuprous oxide were the most efficient. Normally 6—10% of the product was diphenyl ether. This is an equilibrium value, and if such a proportion be added to the reaction mixture previously the whole of the phenyl halide may be recovered as phenol. At 370° diphenyl ether is rapidly hydrolysed by sodium phenoxide solution or other alkali. The hydrolysis of chlorobenzene is effected just as well with sodium carbonate as with sodium hydroxide. In this case there is no need for subsequent acidification. Under reaction conditions either iron, copper, or nickel is appreciably acted on by alkali with generation of hydrogen, and this hydrogen with phenol yields *cyclohexanol*, whence arises loss of product as tar. An iron bomb becomes protected by a coating of magnetic oxide, but any change in heat transmission causes this to flake. Benzenesulphonic acid and sodium hydroxide solution undergo a strictly analogous reaction under similar conditions, and the introduction of diphenyl ether has a similar effect upon the yield. The difficulties which have up to the present prevented the operation of a continuous process on these lines are discussed.

C. IRWIN.

Phosgene. M. and L. JACQUÉ (Chim. et Ind., 1928, 19, 24—38).—Phosgene is universally manufactured to-day by passing a mixture of chlorine with a slight excess of carbon monoxide over activated carbon. External cooling is necessary; the optimum temperature is 125°, and 150° must not be exceeded. Atmospheric oxygen if present results in a loss of yield. Carbon monoxide is prepared by the action of carbon dioxide and oxygen alternately on good quality anthracite or by passing carbon dioxide over heated wood charcoal. It must be freed from dust, carbon dioxide, and moisture. The gases are mixed in lead cylinders fitted with diaphragms and pass to the catalyst chambers, which are also of lead. In the usual process, in which a 37% excess of carbon monoxide suffices, 90% of the phosgene is condensed direct and the remainder absorbed in tetrachloroethane. If oxygen is dispensed with in the manufacture of carbon monoxide a 30% excess of the latter is necessary, and a large quantity of tetra-

chloroethane must be maintained for phosgene absorption with increase both in working losses and in the size of the apparatus. The design of a plant to produce 50 tons of phosgene per week, working with oxygen, is discussed in detail. Small gas generators fed with coke and fired by a current of oxygen with or without admixture of carbon dioxide are used. The gas exit pipe is water-cooled. The apparatus also includes coke scrubber, centrifugal dust separator, and alkaline and acid washers. The activated carbon used in the catalysers gradually deteriorates, but each kg. of carbon should yield at least 2 tons of phosgene. The catalyser proposed consists of horizontal lead cylinders in which the carbon rests on a grid. The exit gases after a preliminary cooling are reduced to -20° by passing through a coil cooled by calcium chloride solution. The unliquified portion passes to a tower in which it is washed with tetrachloroethane. The suitability of a sample of carbon for the process may be judged by determining its power of adsorbing the gases or phosgene. Phosgene is conveniently determined by passing the gas through a saturated aqueous solution of aniline, when crystals of diphenylurea which are almost insoluble in cold water are produced. A concentration of 45 mg./m.³ of phosgene in the atmosphere is dangerous. It can be removed by a spray of caustic alkali or of hexamethylenetetramine. The latter is also suitable for use in gas-masks.

C. IRWIN.

Guaiacolsulphonic acid. TURSKI and others. **Determination of aldehydes.** RADCLIFFE and SWANN.—See XX.

PATENTS.

Manufacture of formaldehyde. FABR. DE PROD. CHIM. "KALA" SOC. ANON., and N. KUARTSCHEV (F.P. 620,754, 7.8.26).—Methyl alcohol vapour and air are led over red-hot carbon. The catalyst may be externally heated or may be mixed with sufficient active carbon to heat it to redness on admission of the reaction mixture. Methyl alcohol which contains acetone or pyridine bases may be employed.

C. HOLLINS.

Manufacture of oxygen-containing carbon compounds. BRIT. CELANESE, LTD., H. DREYFUS, and W. VADER (B.P. 283,989, 20.7.26).—Methyl alcohol or a substance capable of generating it (such as dimethyl ether or methyl formate) is treated with carbon monoxide with or without hydrogen at 300–400° preferably under pressure and in presence of water vapour in contact with an inorganic acid or acid salt, especially phosphoric acid or aluminium phosphate ($\text{Al}_2\text{O}_3 \cdot 12\text{H}_3\text{PO}_4$). Acetic acid or methyl acetate is obtained, and may respectively be converted into acetone or hydrolysed to acetic acid. The methyl alcohol may be prepared catalytically from carbon monoxide and hydrogen, the crude gaseous product being applied. The process may be extended to the manufacture of aliphatic acids (or esters) generally from the next lower alcohols.

C. HOLLINS.

Manufacture of lead alkyls. M. A. YOUTZ, Assr. to GEN. MOTORS CORP. (U.S.P. 1,658,544, 7.2.28. Appl., 7.9.23).—Lead tetra-alkyls are prepared by reducing with nascent hydrogen a mixture of lead and an alkyl chloride.

B. FULLMAN.

Manufacture of aliphatic secondary amino-

alcohols. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of H. SCHOTTE (G.P. 442,413, 25.3.25).—Amino-alcohols, $\text{R} \cdot \text{NH} \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{OH}$, are obtained by the action of alkali on β -chlorourethanes, $\text{R} \cdot \text{NH} \cdot \text{CO}_2 \cdot \text{CH}_2 \cdot \text{CH}_2\text{Cl}$, an oxazolidone being probably formed intermediately. β -Chloroethyl methylcarbamate, b.p. 110–112°/15 mm., obtained from β -chloroethyl chloroformate and methylamine, gives β -methylaminoethyl alcohol, b.p. 159°/750 mm. (picrate, m.p. 148–150°). β -Chloroethyl ethylcarbamate, b.p. 110–111°/12 mm., β -chloroethyl isomylcarbamate, b.p. 106°/1.5 mm., β -chloroethyl benzylcarbamate, b.p. 105°/0.8 mm., or 218–220°/15 mm., β -ethylaminoethyl alcohol, b.p. 169–170° (picrate, m.p. 125–126°), β -isomylaminoethyl alcohol, b.p. 105–106°/15 mm. (picrate, m.p. 94–95°), and β -benzylaminoethyl alcohol, b.p. 148–149°/13 mm. or 105°/1 mm. [picrate, m.p. 104–106° (+ H_2O) or 134–135° (anhyd.)], are also described.

C. HOLLINS.

Treatment [concentration] of [dilute] lower aliphatic acids. BRIT. CELANESE, LTD., H. DREYFUS, and C. I. HANEY (B.P. 283,702, 30.11.26).—Dilute acetic acid (etc.) is extracted with a mixture of a hydrocarbon (e.g., light petroleum, b.p. 40–70°, petrol, b.p. 70–90°, or benzene) and a solvent for the acid (e.g., ether, chloroform, acetone oils), both of lower b.p. than the acid. The extracting mixture may be used in vapour form, and the acid is recovered by distilling off the solvent.

C. HOLLINS.

Preparation of pure lactic acid. C. H. BOEHRINGER SOHN (G.P. 444,956, 27.3.21).—Crude calcium lactate is treated with kieserite (in place of magnesium sulphate crystals, to avoid dilution), and the resulting magnesium lactate is converted directly into lactic acid or, better, since magnesium sulphate is rather soluble in concentrated lactic acid, into calcium lactate by stirring with milk of lime. In the latter case the precipitated magnesium hydroxide carries down coloured and odorous impurities.

C. HOLLINS.

Saponification of esters of organic acids. BRIT. CELANESE, LTD., and W. BADER (B.P. 284,532, 20.7.26).—Organic acids are obtained in concentrated form by hydrolysis of esters with the theoretical amount of water in presence of mineral acid, particularly sulphuric or phosphoric acid, at 150–300°. Ortho- or pyrophosphoric acid contains sufficient water for hydrolysis (with conversion into metaphosphoric acid), and the process may be made continuous, ester and water in theoretical proportions being added to the metaphosphoric acid residue. E.g., methyl acetate vapour and steam are introduced near the bottom of a cylindrical copper vessel, fitted with stirrer and filled with phosphoric acid at 180–220°; glacial acetic acid distils from the vessel (with a little unchanged ester) and the escaping gas is pure dimethyl ether according to the equation: $2\text{Me} \cdot \text{CO}_2\text{Me} + \text{H}_2\text{O} = 2\text{Me} \cdot \text{CO}_2\text{H} + \text{Me}_2\text{O}$. Methyl acetate vapour may also be passed up a tower with copper plates against a counter-current of phosphoric acid which is circulated and hydrated by a steam jet.

C. HOLLINS.

Production of sulphonic acids [from neutral low-temperature tar oils]. GELSENKIRCHENER BERGWERKS-A.-G., and F. SCHÜTZ (G.P. 445,645, 20.9.22).—

Products suitable for use, *e.g.*, as disinfectants, as frothing agents, and for impregnating wood, are obtained by sulphonating neutral low-temperature tar oils. Examples are given of the production of sulphonic acids from oils of b.p. 111° and b.p. 80°. L. A. COLES.

Preparation of cresols and other hydroxylated benzene compounds from creosote liquors. BRAUNKOHLER-PROD. A.-G. (G.P. 444,971, 26.3.24).—Creosotes are distilled over magnesium or aluminium or their alloys (particularly magnalium or electron metal) at 500–600° to give cresols and xlenols. *E.g.*, a crude lignite tar, with 30% of benzine asphalt, distilled with aluminium turnings at 560–580° gave fractions (a) b.p. below 210°, (b) b.p. 210–280°. Alkali extracted 10% of (a), consisting of *o*-, *m*-, and *p*-cresols, and 15–20% of (b), consisting mainly of xlenols. C. HOLLINS.

Manufacture of highly chlorinated paraffins. A. SCHAARSCHMIDT (G.P. 442,829, 27.3.20).—Molten paraffin is chlorinated above 150° until the chlorine content exceeds 61.4%. At 150–160° a yellowish transparent mass is obtained. If this is heated at 180–200° hydrogen chloride is evolved, and further treatment with chlorine at 220° gives a brown, hard mass of high m.p. Free chlorine and hydrogen chloride are removed by bubbling air through the mass. The products serve for manufacture of lacquers, non-inflammable films, and plastic masses. C. HOLLINS.

Chlorination of saturated hydrocarbons. E. KRAUSE and K. ROKA, Assrs. to HOLZVERKOHLUNGS-IND. A.-G. (U.S.P. 1,654,821, 3.1.28. Appl., 4.6.24. Ger., 14.11.23. Cf. U.S.P. 1,591,984; B., 1926, 898).—The process of the patent cited is modified in that hydrogen chloride is used in place of chlorine. T. S. WHEELER.

Manufacture of hydrogenated compounds. J. D. RIEDEL. A.-G. (G.P. 444,665, 19.1.19. Addn. to G.P. 369,374; B., 1923, 543 A).—The catalysts of the prior patent, with the exception of mixed elementary metals, are used for reduction of phenols, aldehydes, and ketones. *E.g.*, mixed carbonates of nickel, cobalt, and copper in atomic proportion, 60:30:10, at 130–170°, catalyse the conversion of benzaldehyde into benzyl alcohol by hydrogen at 25 atm.; at 210–220° cyclohexylmethylcarbinol is obtained in good yield without formation of benzhydrol. Acetone with hydrogen at 15 atm. gives at 100° in presence of the same catalyst isopropyl alcohol; β -naphthol is similarly converted by hydrogen at 190–200° and 10–30 atm. in 3 hrs. into tetrahydro- and in 5 hrs. into decahydro- β -naphthol. cycloHexanol is obtained from phenol and hydrogen at 190–215° and 25 atm. in presence of mixed oxides of the same metals. Cinnthaldehyde with hydrogen under pressure at 140° yields heptyl alcohol, b.p. 174–178°, and some diheptyl ether, b.p. 145°/9 mm. C. HOLLINS.

Catalytic decomposition of cyclic compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 283,600, 12.7.26).—Simple benzene hydrocarbons are obtained by passing more complex compounds with hydrogen at 300–550° over mixed dehydrogenating catalysts, especially those containing iron, cobalt, nickel, molybdenum, vanadium, tungsten, manganese, chromium, or their compounds, activated by admixture

with difficultly reducible oxides of metals. Carbazole, passed with hydrogen at 350° over alumina and iron oxide, gives benzene and ammonia, with some aniline; cresol gives benzene and toluene; naphthalene gives benzene. Similarly, mixed benzene hydrocarbons are obtained from low-temperature tar of b.p. 180–250°.

C. HOLLINS.

3'-Nitro-4'-hydroxy-o-benzoylbenzoic acid. 3'-Amino-4'-hydroxy-o-benzoylbenzoic acid. 4'-Hydroxy-o-benzoylbenzoic acid. 4'-Amino-o-benzoylbenzoic acid. [A, B, C] I. GUBELMANN, II. J. WEILAND, and O. STALLMANN, and [D] I. GUBELMANN, Assrs. to NEWPORT Co. (U.S.P. 1,654,287–1,654,290, 27.12.27. Appl., [A, B] 28.6.26, [C] 29.11.26, [D] 8.12.26).—(A) 3'-Nitro-4'-hydroxy-o-benzoylbenzoic acid, from the corresponding chloro-compound by treatment with boiling aqueous sodium hydroxide solution, has m.p. 175°. (B) 3'-Amino-4'-hydroxy-o-benzoylbenzoic acid is formed from the nitro-compound (A) by reduction. (C) 4'-Hydroxy-o-benzoylbenzoic acid, from the corresponding chloro-compound by treatment with sodium hydroxide solution under pressure at 180° for 12 hrs., has m.p. 212–213°. (D) 4'-Amino-o-benzoylbenzoic acid, from the corresponding chloro-compound by treatment with ammonia solution in presence of copper or of a copper salt under pressure at 180° for 18 hrs., has m.p. 200° [decomp.] (acetyl derivative, m.p. 277°).

T. S. WHEELER.

Manufacture of condensation products of arylamines. P. HALLER and H. KAPPELER (B.P. 266,358, 17.2.27. Switz., 17.2.26).—An arylamine (aniline, toluidine) is condensed in an acid medium with formaldehyde below 100°, and the orange-red product is washed and treated with alkali (solution of sodium hydroxide, ammonia, lime, sodium carbonate, or sulphide) to give a bone- or horn-substitute. Alternatively, the base and formaldehyde are condensed first in alkaline medium and the product is condensed with more formaldehyde in acid medium, followed by washing and treatment with alkali. Modified products are obtained by adding to the reaction mixture a diluent (water, glycerol, acetone, etc.), a filler (carbamide, tannin, cellulose, leather powder, asbestos, calcium phosphate, barium sulphate, emery, etc.), or a pigment (indigo, nigrosine, chrome yellow, etc.). The final product may further be heated above 100° with or without linseed oil etc. C. HOLLINS.

Manufacture of isatins and N-arylsulphonyl derivatives thereof. I. G. FARBENIND. A.-G. (B.P. 265,224, 28.1.27. Ger., 28.1.26).—Arylsulphonyl amides in the form of alkali salts are condensed with oxalyl chloride in a solvent (carbon disulphide) and the products are cyclised by means of aluminium chloride etc. to give N-arylsulphonylisatins, from which isatins are obtained by hydrolysis with alkali or cold concentrated sulphuric acid. N-*p*-Toluenesulphonylisatins are thus prepared from the *p*-toluenesulphonyl derivatives of *p*-toluidine (m.p. 202–205°), *m*-toluidine (mixture, m.p. 161–163°), and 3-chloro-*p*-toluidine (m.p. 180–190°); α -naphthylamine gives 1:8-naphthisatin, m.p. above 300°, instead of the expected 1:2-compound. Sodium *p*-toluenesulphon-3-chloro-*p*-toluidide, C₇H₇·SO₂·NNa·C₆H₄Cl, has m.p. 141–142°. C. HOLLINS.

Manufacture of 4-nitrobenzthiazoles. I. G. FARBENIND. A.-G., Assees. of K. BRAND (G.P. 442,773, 8.7.24).—Acylated 2-chloro-5-nitroanilines are treated with sodium sulphide (or with sodium disulphide, followed by reduction of the disulphide to a thiophenol) and then acidified to give 4-nitrobenzthiazoles. Examples are: 4-nitrobenzthiazole from 2-chloro-5-nitroformanilide, and its 1-methyl (m.p. 136–137°) and 1-phenyl (m.p. 192°) derivatives from the corresponding acetanilide and benzanilide, respectively. C. HOLLINS.

Manufacture of indophenols. I. G. FARBENIND. A.-G., Assees. of F. BALLAUF (G.P. 443,685, 3.11.25).—Indophenols are obtained from dihydroindoles by the usual general methods, and may, if desired, be reduced to leuco-indophenols. The products are intermediates for sulphide and vat dyes. 2-Methyldihydroindole is oxidised with chromic acid in presence of *p*-aminophenol, or is condensed with quinonechloroimide in concentrated sulphuric acid at 10°, and the product, after reduction to the leuco-indophenol, is made acid to Congo paper and salted out. Dihydroindole, 2-phenyldihydroindole, and 1:2-dimethyldihydroindole are similarly oxidised with *p*-aminophenol, and 2-methyldihydroindole with 2:6-dichloro-4-aminophenol. C. HOLLINS.

Manufacture of chlorine-substitution products of 1-amino-2:4-dimethylbenzene [chlorinated *m*-4-xyldines]. I. G. FARBENIND. A.-G. (B.P. 278,761, 11.10.27. Ger., 11.10.26).—4-Nitro-*m*-xylene is chlorinated in carbon tetrachloride solution in presence of ferric chloride, giving at 0° crystals of 2-chloro- and an oily 6-chloro-derivative, which are reduced to 2-chloro-*m*-4-xyldine, m.p. 47–48°, and 6-chloro-*m*-4-xyldine, m.p. 96–97°, respectively. Further chlorination, or chlorination without cooling in ice, gives 2:6-dichloro-4-nitro-*m*-xylene, reducible to 2:6-dichloro-*m*-4-xyldine, m.p. 56–57°. The bases are volatile in steam. C. HOLLINS.

Oxidation of arylalkyldithiocarbamic acids to the corresponding disulphides. SILESIA VER. CHEM. FABR. (B.P. 279,790, 11.7.27. Ger., 30.10.26).—The dithiocarbamates obtained by interaction of carbon disulphide and alkylarylamines are oxidised in alkaline solution to the thiuram disulphides by a current of nitric oxide mixed with air. C. HOLLINS.

Manufacture of pure urea. L. BUB, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,659,190, 14.2.28. Appl., 9.11.25. Ger., 15.11.24).—See B.P. 249,041; B., 1926, 420.

Manufacture of sulphuric acid esters of aromatic hydroxyalkyl ethers. W. HENTRICH and M. HARTMANN, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,644,524, 4.10.27. Appl., 21.6.26. Ger., 25.6.23).—See B.P. 266,940; B., 1927, 324.

Catalysts (B.P. 281,218).—See X. Detergents (B.P. 284,367).—See XII. Mercaptobenzthiazoles (B.P. 283,661 and 283,679).—See XIV.

IV.—DYESTUFFS.

Oxidation of organic dyestuffs and of cellulose on exposure to light. V. V. SCHARVIN and A. B. PAK-

SCHVER (J. Russ. Phys. Chem. Soc., 1927, 59, 459–463).—See B., 1927, 837.

Arsenic in coated papers etc. STERN.—See V.

PATENTS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G. (B.P. 265,232 and 265,964, [A] 29.1.27, [B] 8.2.27. Ger., [A] 29.1.26, [B] 9.2.26. Addn. [A, B] to B.P. 237,294; B., 1925, 840).—The dyes of the prior patent are obtained by condensing 1:4:5:8-naphthalene-tetracarboxylic acid or its anhydride with (A) an *o*-nitroarylamine, and reducing the di-*o*-nitroarylimide so formed, whereby 3-nitro-*p*-phenetidine gives a brown vat dye, *o*-nitroaniline a red; or with (B) an *o*-diamine in alcohol or other low-boiling solvent, and heating the resulting product in acetic acid or other high-boiling solvent. C. HOLLINS.

Production of benzantrones and their derivatives. BRIT. ALIZARINE CO., LTD., W. H. DAWSON, C. W. SOUTAR, and J. ANDERSON (B.P. 284,035, 3.11.26).—Glycerol is treated with thionyl chloride, sulphuryl chloride, or sulphur mono- or di-chloride, preferably in equimolecular quantities, and the reaction mixture is then condensed in the usual way with anthranol and sulphuric acid to give a benzanthrone. Alkaline fusion of the product obtained by using thionyl chloride gives a bright blue vat dye. When sulphur dichloride is used a benzanthrone containing sulphur results. By using sulphur monochloride, sulphur is introduced in the Bz-ring and a dibenzanthronyl is obtained by alkaline fusion of the product. C. HOLLINS.

Manufacture of vat dyes of the dibenzanthrone series. I. G. FARBENIND. A.-G. (G.P. 443,610, 24.3.23).—The hydroxylated dibenzanthrones of G.P. 414,203 and 414,924 (B., 1925, 799; 1926, 234) are alkylated or arylated. By the action of methyl *p*-toluenesulphonate on tetrahydroxydibenzanthrone a violet vat dye is obtained, on dihydroxydibenzanthrone a grey-blue vat dye. C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 260,588, 25.10.26. Ger., 29.10.25).—Anthraquinone-acridones (B.P. 894 of 1911) are obtained in a single operation by heating 1-chloroanthraquinone-2-carboxylic acid with an arylamine (β -naphthylamine, *p*-chloroaniline), preferably in presence of a mild condensing agent (borax, potassium hydrogen sulphate, boric acid, oxalic acid, arsenious oxide, sodium acetate with boric acid, etc.), in a solvent (nitrobenzene, trichlorobenzene). C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 263,494, 21.12.26. Ger., 22.12.25).—The alkylation products of pyrazolanthrones give by alkaline fusion red vat dyes. C. HOLLINS.

Manufacture of dyes of the anthanthrone series. L. CASSELLA & Co., G.M.B.H. (B.P. 260,998 and Addn. B.P. 280,217, [A] 3.11.26, [B] 2.11.27. Ger., [A] 7.11.25, Switz., [B] 2.11.26).—(A) Anthanthrone (G.P. 280,787) is halogenated under milder conditions than those described in G.P. 287,250 (B., 1916, 354). *E.g.*, chlorine in trichlorobenzene at 150°, or in sulphuryl chloride at 60° in presence of iodine, gives a yellowish-orange

vat dye; bromine in nitrobenzene at 160°, or in concentrated sulphuric acid at 50–100° in presence of iodine, gives a reddish-orange vat dye. (B) The dyes of the prior patent are obtained by cyclisation of 1:1'-dinaphthyl-8:8'-dicarboxylic acid in sulphuric acid followed by halogenation in the same medium without isolation. Economy of bromine may be effected by adding sufficient sulphur trioxide (as high-strength oleum) to oxidise the hydrogen bromide, before bromination.

C. HOLLINS.

Preparation of condensation products [vat dyes] of the anthraquinone series. I. G. FARBENIND. A.-G., Assees. of H. TESCHE and P. TUST (G.P. 443,584, 14.2.25).—*o*-Diaminoanthraquinones are heated with metal salts or oxides, preferably in a solvent. 1:2:4-Triaminoanthraquinone heated with ferric chloride in nitrobenzene with addition of sodium acetate yields a brown to brown-black vat dye, also obtained by using copper chloride or by using red lead in acetic acid, or by the action of hydrochloric acid or sodium hydroxide on the intermediate product prepared by action of copper chloride and sodium acetate on the triaminoanthraquinone in acetic acid. 1:2-Diamino-4-benzamidoanthraquinone is similarly converted into a red-brown vat dye, 1:2-diaminoanthraquinone a brown, and 2:3-diaminoanthraquinone a brown-olive.

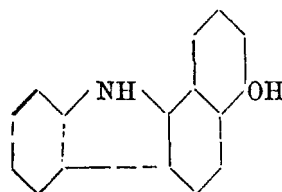
C. HOLLINS.

Purification of vat dyes. SOC. CHEM. IND. IN BASLE [GES. F. CHEM. IND. IN BASEL] (B.P. 278,728, 6.10.27. Switz., 6.10.26).—The vat dyes made from aminoanthraquinones and cyanuric chloride etc. (B.P. 205,525 [see U.S.P. 1,437,783], 231,688, 234,086, 237,872; B., 1923, 490 A; 1925, 438, 624, 839) are improved in purity and fastness by treatment with aqueous hypochlorite solutions.

C. HOLLINS.

Vat dyes of the isatin-oxythionaphthen group. C. KRAUHS, Assr. to GRASSELLI DYE STUFF CORP. (U.S.P. 1,650,139, 22.11.27. Appl., 5.1.26. Ger., 8.1.25).—7-Methylisatin or a chloro-derivative is condensed with an oxythionaphthen to give dyes yielding in the vat yellow to orange to brown shades. T. S. WHEELER.

Manufacture of indigoid vat dyes. KALLE & Co. A.-G., Assees. of M. P. SCHMIDT and O. HERRMANN (G.P. 441,439, 27.9.24).—An isatin α -anil or α -chloride is condensed with a tetrahydronaphthacarbazole, with subsequent halogenation if desired. *E.g.*, 4-hydroxy-7:8:9:10-tetrahydro- α -naphthacarbazole (as annexed formula), prepared by alkaline



fusion of the product from cyclohexanone and α -naphthylhydrazine-5-sulphonic acid, gives with 5-chloroisatin α -anil a violet vat dye; 3-hydroxy-7:8:9:10-tetrahydro- β -naphthacarbazole with isatin α -anil a blue; 3-hydroxy-8(?)-methyl-7:8:9:10-tetrahydro- β -naphthacarbazole with isatin α -anil a blue-violet.

C. HOLLINS.

Manufacture of azo dyes. I. G. FARBENIND. A.-G., Assees. of FARBENFABR. VORM. F. BAYER & Co. (B.P. 259,970, 13.10.26. Ger., 14.10.25).—Direct developing

green dyes of the type *p*-diamine \rightarrow middle component \rightarrow an aminonaphtholsulphonic acid carrying an external amino-group \rightarrow 2-methylindole or 1-phenyl-3-methylpyrazolone, are described. The *p*-diamine may be *p*-nitroaniline-*o*-sulphonic acid (reduced after final coupling), or *p*-aminoacetanilide-3-sulphonic acid or other acylated *p*-diamines of the benzene or naphthalene series (hydrolysed after final coupling), or derivatives of these. As middle components are used cresidine, α -naphthylamine, α -naphthylamine-6(or 7)-sulphonic acid or its 2-ethoxy-derivative, 1-amino-5-naphthol-7-sulphonic acid, 1-amino-2-naphthol ethers, etc., a middle component of the benzene series or of the naphthalene series being employed with a first component of the same series. Suitable third components with an external amino-group are the *p*-amino benzoyl derivatives of 2:5:7-, 2:8:6-, or 1:8:4-aminonaphtholsulphonic acids, or the naphthiminazoles containing an aminophenyl substituent in the hetero-ring, derived from 1:2:5:7-, 1:2:8:6-, and 1:2:8:4-diaminonaphtholsulphonic acids, and the corresponding naphthathiazoles. The dyes may be diazotised and developed on the fibre with β -naphthol or 1-phenyl-3-methylpyrazolone.

C. HOLLINS.

Manufacture of azo dyes. I. G. FARBENIND. A.-G. (B.P. 263,164, 15.12.26. Ger., 15.12.25).—Diazo compounds are coupled with arylamides of sulphonated 2:3-hydroxynaphthoic acid (B.P. 183,428; B., 1922, 853 A) in substance or on the fibre, the process of B.P. 260,339 (B., 1927, 9) being excluded. The azo dyes so formed dye animal or vegetable fibres or may be used for lake-formation. Examples are: sulphonated 2:3-hydroxynaphthoic anilide with diazotised aniline (orange), *m*-aminobenzanilide (yellowish-red), *m*-aminobenzaldehyde (yellowish-red), or aniline-*o*-sulphonic acid (red); *o*-toluidide or 4-chloro-*o*-aniside with diazotised *o*-phenetidine or *m*-4-xylydine (bluish-red); *o*-toluidide with tetrazotised dianisidine (bluish-violet); 4-chloro-*o*-toluidide with diazotised 4-nitro-*o*-toluidine (ruby-red); β -naphthylamide with *o*-phenetoleazo- α -naphthylamine (black); and sulphonated bis-2:3-hydroxynaphtholylbenzidine with diazotised *p*-nitroaniline (brownish-red ice colour on cotton).

C. HOLLINS.

Manufacture of [azo] dyes containing chromium. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 283,979, 17.7.26).—Azo dyes, made by coupling a diazotised *o*-aminophenol with a naphthol or an arylaminonaphtholsulphonic acid, are heated with a chromium solution under pressure, with or without subsequent treatment with alkali. The chromium compounds of (a) 1:2:4-aminonaphtholsulphonic acid \rightarrow β -naphthol and (b) *p*-nitro-*o*-aminophenol \rightarrow 2-phenylamino-8-naphthol-6-sulphonic acid, dye wool in pure blue and dark brown shades, respectively.

C. HOLLINS.

Manufacture of black copying colours. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 283,777, 24.5.27).—Black copying colours are made by coupling a diazotised dialkylated phenosafranine with a cresol, particularly diethylphenosafranine with *p*-cresol (cf. B.P. 14,687 of 1895).

C. HOLLINS.

Chromiable brown disazo [mordant] dyes. W. NEELMEIER, T. NOCKEN, and W. REBNER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,643,222, 20.9.27. Appl., 22.4.26. Ger., 28.4.25).—See B.P. 251,637; B., 1927, 771.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Chemistry of *Phormium tenax*. P. W. AITKEN (New Zealand J. Sci. Tech., 1927, 9, 226—228).—The present state of knowledge of the constituents of *Phormium tenax* (New Zealand flax) is summarised.

J. R. NICHOLLS.

Manufacture of ground-wood pulp. H. NAKAMURA and Y. SASAKA (Sexagint [Osaka celebration], Kyoto, 1927, 181—184).—Analyses of ground-wood pulp coming directly from the stone and of that collected on the centrifugal pulp screens show the latter to contain more cellulose and less pentosan than the former.

H. F. GILLBE.

Arsenic in coated papers and boards. H. J. STERN (Analyst, 1928, 53, 83—86).—A paper or board used for wrapping in its uncoated state is not likely to contain any dangerous amount of arsenic, nor are the adhesives employed for coating sources of danger. Of the synthetic pigments used, antimony oxide may be employed as part of the base for colour lakes, and the so-called arsenical greens may contain over 30% As_2O_3 . There appears to be no danger from the lakes of natural dyewoods, and, except for dyestuffs (such as Magenta and Methyl Violet) precipitated by arsenious oxide in alkali solution, or those produced by means of tartar emetic, synthetic dyestuffs and pigments are generally free from arsenic, although contamination during manufacturing processes is possible. Pigment Scarlet 3B and Orange II quite commonly contain 50—100 pts. of arsenic per million, and Pigment Fast Red PRL, Fast Orange RL, and colours of the type of Patent Blue A (Disulphine Blue A) are also liable to contain arsenic. A limit for arsenic in coated boards and papers in force with certain users is 10 pts. per million.

D. G. HEWER.

PATENTS.

Preparation of fibrous vegetable materials for textile and other purposes. VICKERS, LTD., and O. D. LUCAS (B.P. 283,285, 8.10.26, 4.12.26, and 2.4.27).—The material, after scutching, is boiled first with caustic soda solution of less than 2% strength and then, with or without washing, with a similar solution of alkali containing 0.1% of an oil such as linseed oil, suitable times of treatment for retted flax being 1 hr. and 0.25 hr. respectively. After washing with cold water the fibres are treated with weak acid, e.g., 0.2—0.4% hydrochloric acid, optionally containing a small proportion of sodium hypochlorite, and, after further washing, are dried. The operations are preferably carried out in open vats with the fibres loosely arranged in reticulated trays, to which latter a vertical reciprocating movement is given to ensure efficient circulation of the treating liquor.

D. J. NORMAN.

Improvement of half-wool fibrous material which cannot be felted. A. S. KESSLER (G.P. 446,267, 7.4.26).—Low-grade material containing an excess of

cotton is treated cold with strong alkali solutions containing strengthening and moistening agents, e.g., glycerin, alcohol, sulphite-cellulose waste liquor, etc., as well as material for protecting the wool.

L. A. COLES.

Improvement of cloth by the galvanic deposition of metals upon it. W. ENZ (Austr. P. 106,449, 11.1.26. Ger., 15.1.25).—The cloth during the electrolytic treatment is tightly packed on an electrode constructed of plastic, conducting or partially conducting material surrounding a solid core; e.g., a mixture of freshly-precipitated copper sulphide and oleic acid packed around a sheet of copper is used for coppering the cloth.

L. A. COLES.

Impregnation of fibres and fibrous materials. P. HEERMANN (G.P. 445,771, 18.5.26).—Heterocyclic bases, such as pyridine and its derivatives, are used as solvents in impregnating fibrous materials with oleates, stearates, resinates, etc. in water.

L. A. COLES.

Removal of fat from raw sheep's wool. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 282,164, 17.9.26).—The organic esters of carbonic acid, particularly the methyl and ethyl esters, are used, either alone or in conjunction with the more usual reagents, for extracting fat from raw wool. These esters differ from other solvents in that they do not cause twisting and tangling of the wool nor do they remove dirt. The solvent adhering to the wool may be washed out with water and recovered by extracting the aqueous liquor with a suitable solvent, e.g., dichlorobenzene.

D. J. NORMAN.

Treatment of fibrous material to render it proof against moth. L. E. JACKSON and H. E. WASSELL (B.P. 263,092, 29.9.26. U.S., 17.12.25; cf. B., 1927, 870).—The material is treated with a cinchona alkaloid or a derivative thereof. Quinidine salts are particularly suitable, and may be applied in solution in water (as hydrochloride), alcohol (as sulphate), or petroleum naphtha or other organic solvent (as oleate). The strength of the solution should be at least 1% calculated as quinidine. [Stat. ref. to B.P. 146,225, 104,684, and 8858 of 1895.]

D. J. NORMAN.

Moth-proofing compounds. I. G. FARBENIND. A.-G. (G.P. 442,901, 17.7.25).—Phthalic acid and its derivatives, especially neutral or acid phthalic esters, phthalimide, halogenophthalic esters or acids, hydroxy, phthalic acids, methylamine and pyridine phthalates are claimed as moth-proofing agents. They may be used as solids, or in solution in volatile solvents. The butyl and amyl esters are specially mentioned.

C. HOLLINS.

Production of figures on vegetable textile material by mercerisation. E. GMINDER (B.P. 267,566, 14.3.27. Ger., 13.3.26).—The fabric is impregnated with a mercerising solution, stretched, and printed while under tension with a suitable neutralising medium, e.g., dilute hydrochloric acid in the case of alkaline mercerising agents. The printed fabric is then allowed to shrink.

D. J. NORMAN.

Treatment [weighting] of yarns, fabrics, films, etc. [containing cellulose esters or ethers]. H. DREYFUS (B.P. 281,084, 26.5.26).—Increased quantities

of loading agents can be introduced into cellulose esters or ethers if the material is treated with aqueous solutions of swelling agents, particularly organic swelling agents, *e.g.*, glycollic acid, lactic acid, etc., before or during each stage of the loading operation or repetition thereof. Suitable swelling solutions are aqueous solutions containing 7–10% of acetone, 50% of ethyl alcohol, 15–20% of diacetone alcohol, or 10–12% of ammonium thiocyanate. D. J. NORMAN.

Manufacture of artificial [silk] threads. L. LILIENFELD (B.P. 281,352, 30.6.26).—The extensibility of artificial threads produced by the process described in B.P. 274,521, 274,690 (B., 1927, 745), and 264,161 can be increased to at least 8% without deleteriously affecting the strength or lustre by treating the threads with shrinking agents under conditions which permit complete or partial shrinkage to occur. Suitable shrinking agents include mercerising solutions, *e.g.*, caustic soda solutions (18–25%) at 15–18° with or without the addition of neutral or alkaline salts or mono- or poly-hydric alcohols, sodium sulphide solution (50–58%), and mixtures of caustic soda and sodium sulphide. If desired the threads may be pretreated with protective agents such as starch, gelatin, etc. The mercerised threads are centrifuged, transferred (with or without washing) to a neutralising bath, washed, bleached, and finally dried without tension. The process is also applicable to artificial silk in fabric form, and to mixtures of artificial silk with vegetable or animal fibres. D. J. NORMAN.

Manufacture of threads, filaments, etc. from viscose. COURTAULDS, LTD., H. J. HEGAN, and E. HAZELEY (B.P. 282,973, 20.12.26).—Filaments which show a subdued lustre and a certain roughness of surface resulting in improved felting properties are made by spinning a viscose solution containing a small proportion of added soluble carbonate (2–4%) and sufficient caustic soda, *e.g.*, over 7%, to give a ratio NaOH:CO₂ of, *e.g.*, 4:1 or more into a sulphuric acid bath containing a high proportion, *e.g.*, 25% or more, of sodium sulphate. The filaments show little inflation as evidenced by their density (1.45–1.53). D. J. NORMAN.

Manufacture of artificial materials from viscose. L. LILIENFELD (B.P. 281,351, 29.6.26. Addn. to B.P. 274,521; B., 1927, 745).—Artificial threads having a strength of more than 2 g. per denier in the dry state are made by spinning a viscose solution containing not more than 5% of caustic alkali calculated as NaOH into a bath containing 45–55% of sulphuric acid, as H₂SO₄. D. J. NORMAN.

Production of alkali-cellulose from sheets of cellulose impregnated with alkali solution. I. G. FARBENIND. A.-G. (G.P. 445,728, 27.5.24).—The vats in which the sheets of cellulose have been steeped are drained and tilted until the sheets, which during the steeping hang vertically, lie in a horizontal or nearly horizontal position. Gentle pressure is applied to the sheets by means of partition walls which slide in grooves in the sides of the vat, thus expressing the greater part of the excess liquor, the remainder being subsequently removed in presses. L. A. COLES.

Treatment of natural and artificial cellulose fibres with alkali. CHEM. FABR. VORM. SANDOZ (B.P.

279,784, 10.6.27. Ger., 29.10.26).—Raw and sized cotton fabrics may be satisfactorily mercerised without a preliminary bowking if a small quantity (1–2%) of a suitable mixture of phenols (particularly the cresols and higher homologues) and hydrogenated aromatic compounds, *e.g.*, tetrahydronaphthalene, cyclohexanol, decahydro- β -naphthol, is added to the mercerising liquor. Mixtures containing 88–98% of phenols and 12–2% of a hydroaromatic compound give particularly good results. D. J. NORMAN.

Treatment of precipitated acetylcellulose. VER. F. CHEM. IND. A.-G. (B.P. 269,543, 12.4.27. Ger., 15.4.26).—The precipitated cellulose acetate is formed into a continuous coherent web, and, after squeezing between heavy rollers to remove as much as possible of the adherent acid liquor, is washed with water in a series of vats working on the counter-current principle. The web is finally dried on drying cylinders. The process is continuous and facilitates the recovery of acetic acid. D. J. NORMAN.

Spinning of artificial threads according to the stretch-spinning process. W. SCHULZ (B.P. 261,365, 4.11.26. Ger., 11.11.25).—During the stretch-spinning of, *e.g.*, cuprammonium hydroxide solutions of cellulose into a bath at about 40°, there is a tendency for the gases initially dissolved in the coagulant to form bubbles which intermingle with the filaments. To avoid this, sufficient pressure is applied to the precipitating liquor to prevent the liberation of gases therefrom at the operating temperature. Thus the liquor may be supplied to the spinning funnel under any desired head of pressure through a pipe, the diameter of which is large compared with that of the outlet of the funnel. D. J. NORMAN.

Treatment of artificial silk yarns after spinning. F. J. GAILBERT (B.P. 283,752, 10.2.27).—Artificial silk yarn, closely resembling natural silk in appearance and capable of giving a solid washable fabric showing no irregularities after dyeing and finishing, is obtained by passing ordinary twisted artificial silk yarn under tension through a bath of, *e.g.*, water, and then imparting to it a further mechanical twist. D. J. NORMAN.

Treatment of rayon [artificial silk]. E. K. GLADDING and T. E. SHARPE, Assrs. to DU PONT RAYON Co., Inc. (U.S.P. 1,655,097, 3.1.28. Appl., 27.3.26).—Artificial silk is freed from copper by treatment with dilute (0.2%) sodium cyanide solution containing 0.1% of ammonia. T. S. WHEELER.

[Weighting of] cellulose [acetate] fibres, fabrics, and articles. BRIT. CELANESE, LTD. (B.P. 258,874, 21.9.26. U.S., 22.9.25).—Fabrics composed of or containing cellulose esters or ethers are impregnated at, *e.g.*, 45–65° with an aqueous solution of a metal salt, which is a swelling agent for the ester or ether, and are then treated with a reagent to precipitate the metal as an insoluble derivative. *E.g.*, 1 pt. of cellulose acetate silk is soaked in 30 pts. of stannic chloride solution (*d* 1.2) for 0.5 hr. at 40–50°, and, after rinsing, is transferred to a solution of disodium hydrogen phosphate (*d* 1.035). After a further rinse the fabric is treated with sodium silicate solution (*d* 1.035), and is finally washed with or without soap solution. Metal salts which are not swelling agents for cellulose acetate may be used in

conjunction with such swelling agents as dilute acetic acid. The treated silk shows not only increased weight and volume, but also an increased resistance to heat.

D. J. NORMAN.

Compositions containing cellulose esters and ethers etc. H. J. HANDS, and SPICERS, LTD. (B.P. 279,139, 21.4.26).—Chloropropanes having more than three atoms of chlorine per mol., *e.g.*, penta-, hexa-, or hepta-chloropropane (including all isomerides) or mixtures of these, are used as plasticising agents for cellulose esters or ethers. When used in small quantities, *e.g.*, 2–3% on the weight of ester, in association with other known plasticising agents, they are effective in reducing inflammability.

D. J. NORMAN.

Treatment of bagasse fibres preparatory to pulp board making. H. T. PRICE (U.S.P. 1,656,829, 17.1.28. Austral., 16.7.26).—The material is suspended in water and heated at 100°. Caustic soda is added and the mixture boiled for about 1 hr. with stirring. The bagasse is then passed between rollers and is again boiled for 1 hr.

D. J. NORMAN.

Drying of waste sulphite liquors and other viscid liquids to obtain dry granular solids. H. G. C. FAIRWEATHER. FROM INDUSTRIAL WASTE PRODUCTS CORP. (B.P. 282,480, 6.9.26).—The heated or superheated liquor is sprayed into the hottest part of a current of heated gas and is carried along therewith through a drying chamber until desiccation is complete. The temperature of the drying gas may be up to 538° at its hottest part, and should at all parts be above the b.p. of the liquor. The separated dry particles have a glazed surface and consequently show a reduced tendency to coalesce. [Stat. ref. to B.P. 215,315, 209,148, 190,099, 151,965, 145,079, and 19,350 of 1913.]

D. J. NORMAN.

Manufacture of transparent papers, particularly those used for the packing of edible articles. O. KLOTZ (B.P. 283,751, 7.2.27).—Pergamyn, parchment, tissue, or other suitable paper is coated on one or both sides with a solution of gelatin.

D. J. NORMAN.

Testing of pulp suspensions. M. O. SCHUR, Assr. to BROWN Co. (U.S.P. 1,653,125, 20.12.27. Appl., 20.6.24).—The suspension is poured on to a foraminous screen and the thickness of the resulting layer of pulp is noted.

D. J. NORMAN.

Continuous manufacture of cellulose acetate. Soc. CHIM. DES USINES DU RHÔNE (B.P. 270,656, 24.3.27. Fr., 10.5.26).—See F.P. 615,879; B., 1927, 811.

Manufacture of [multiple-ply] textile fabrics. E. WEINHEIM (B.P. 255,476, 16.7.26. Ger., 17.7.25).

Production of combed materials, rovings, fine yarns, etc. from artificial fibres. C. NIETHAMMER (B.P. 275,540, 15.10.26. Ger., 9.8.26).

[Apparatus for] impregnating and rendering rigid pieces of paper or fabric of any form. MANUF. DE MACHINES AUXILIAIRES POUR L'ÉLECTRICITÉ ET L'IND. (B.P. 280,386, 10.12.26. Addn. to B.P. 215,726).

Keratin threads (G.P. 445,503).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Development of vat dyes on the fibre by means of nitrites. I. G. FARBENIND. A.-G., Assees. of K. JELLINEK (G.P. 441,984, 20.6.25).—Leuco-vat dyes are oxidised with nitrite in a bath containing formic acid and an electrolyte. This makes possible the use of vat colours with ice-colours for printing. Bright, fast prints are obtained.

C. HOLLINS.

Dyeing of animal fibres, in particular silk, by means of ice-colours. J. W. LEITCH & Co., LTD., A. E. EVEREST, and J. A. WALLWORK (B.P. 283,347, 8.7.26).—Silk or wool, or a mixture of these or a mixture with cotton or viscose, in loose fibre, yarn, or piece goods, is padded with a dilute neutral or alkaline solution of α - or β -naphthol containing soap, Turkey-red oil, etc. and, after squeezing or hydro-extraction, is treated with a diazo solution at 0°, or is printed with a diazo paste. Thus a 2-litre padding bath may contain 1 g. of β -naphthol, 5 g. of olein or stearin soap, and 10 g. of sodium carbonate. The naphthol may with less advantage be applied in dilute aqueous alcohol, acetone, or pyridine solution.

C. HOLLINS.

Dyeing of wool or silk by means of ice-colours. J. W. LEITCH & Co., LTD., A. E. EVEREST, and J. A. WALLWORK (B.P. 283,838—9, 8.7.26).—(A) Wool, alone or in mixtures (except with silk), (B) silk, alone or in mixtures, is dyed by the process described in B.P. 283,347 (preceding), using in place of a naphthol a 2 : 3-hydroxy-naphthoic arylamide.

C. HOLLINS.

Dyeing of cellulose acetate and materials made therefrom. I. G. FARBENIND. A.-G., Assees. of H. KESSELER and E. DÖRING (G.P. 444,961, 22.2.24).—Acetate silk is dyed with basic dyes with addition of not more than 5 g. of a soluble thiocyanate per litre of dye bath, together with the requisite water-soluble colloid. Dyeings fast to rubbing are obtained.

C. HOLLINS.

Dyeing, printing, or stencilling of materials composed of or containing cellulose esters. BRIT. CELANESE, LTD., and G. H. ELLIS (B.P. 284,376, 21.10.26).—The processes for the dyeing etc. of cellulose acetate materials with dispersions of insoluble coloured organic compounds (B.P. 219,349, 224,925, 242,393, 242,711, 269,960, 273,819, 273,820; B., 1924, 906; 1925, 39; 1926, 87, 50; 1927, 475, 650) are extended to the dyeing etc. of materials made from other cellulose esters, except cellulose formate obtained by formylation with strong formic acid and a catalyst below 5° (cf. B.P. 230,650, B., 1927, 103). Examples are: cellulose formate silk dyed with 4-chloro-2-nitro-4'-ethoxydiphenylamine or 5 : 7-dibromo-3-indole-2'-thionaphthene-indigo in presence of ammonium sulphoricinoleate; cellulose propionate silk dyed with 5 : 8-dimethyldiaminoalizarin in presence of sulphonated naphthalene-ricinoleic acid condensation product, or with 4-chloro-2-nitro-4'-methoxydiphenylamine in presence of xylene and Turkey-red oil.

C. HOLLINS.

Printing with vat dyes. I. G. FARBENIND A.-G., Assees. of H. GOSSLER (G.P. 444,962, 26.2.25).—Vat dyes are printed with alkalis and excess of zinc dust (or other metal which develops hydrogen in presence of alkali),

and steamed. The prints before steaming are quite stable, and the shade obtained is fuller than by the usual methods.

C. HOLLINS.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Manufacture of hydrated baryta from the carbonate. P. BAUD (Compt. rend., 1928, 186, 438—441).—A mixture of barium carbonate (100 pts.) and ferric oxide (12—15 pts.) heated in a tube-furnace at 1150—1180° for 1—2 hrs. yielded a black product containing 56—58% BaO (water-soluble). The addition of calcium carbonate rendered the mass porous as a result of the liberation of carbon dioxide, whilst the use of a mineral containing 76% Fe₂O₃ yielded a clinker containing 132% BaO.9H₂O. High yields were obtained on the commercial scale, and the process might be used for the preparation of alkali and barium sulphate: $\text{Ba(OH)}_2 + \text{Na}_2\text{SO}_4 = \text{BaSO}_4 + 2\text{NaOH}$.

J. GRANT.

Phosgene. JACQUÉ.—See III. **Germicidal efficiency of sodium hydroxide.** LEVINE and others. **o-Tolidine test for chlorine.** McCrUMB.—See XXIII.

PATENTS.

Catalytic production of hydrocyanic acid from formamide. I. G. FARBENIND. A.-G. (B.P. 269,166, 1.4.27. Ger., 10.4.26).—Vapours of formamide or formamide containing ammonium formate are highly diluted with ammonia and/or inert gases, and are passed at high velocity at reduced pressure, and above 300°, over solid metals the catalytic action of which is not impaired by the temperature and reaction materials, or are passed through tubes made of, or lined with, such metals. Examples of such catalysts are iron, aluminium, nickel, or V2A steel tubes, alone or filled with various metallic turnings.

W. G. CAREY.

Manufacture of active silica. I. G. FARBENIND. A.-G. (B.P. 255,864, 19.7.26. Ger., 23.7.25).—Silica is precipitated from suitable silicon compounds without any intermediate formation of a sol or gel in an acid, neutral, or only slightly alkaline solution, the precipitate is freed from mother-liquor and subjected to a pressure of 100 atm. or more without heating.

W. G. CAREY.

Effecting [purifying hydrogen by] catalytic reactions for the production of ammonia. LAZOTE, INC., Assees. of R. WILLIAMS (B.P. 258,887, 23.9.26. U.S., 24.9.25).—Hydrogen containing 2—10% CO is passed under a pressure of 900 atm. at 400° over a catalyst made by heating zinc carbonate or a mixture of zinc and chromic hydroxides at 400°. In this way the carbon monoxide is converted into methyl alcohol, which is removed by cooling the gases, still under pressure, and at the same time acts as a solvent for traces of other impurities in the gases. After passing through the cooler, the last traces of water and methyl alcohol are removed by means of active charcoal, and the hydrogen-nitrogen mixture is then passed directly over the ferric oxide catalyst to obtain ammonia.

A. R. POWELL.

Production of salts from brines and solutions. O. V. MARTIN, Assr. to MARTIN-COLVIN CO. (U.S.P. 1,657,633, 31.1.28. Appl., 19.5.26).—The solution is heated and a portion is converted into a mist over the

rest of the solution, thereby warming the air with which the spray comes into intimate contact and keeping it above its dew point. Rapid evaporation of the solution occurs in the zone of mist.

W. G. CAREY.

Manufacture of alkali nitrates. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 283,771—2, [A] 25.4.27, [U] 29.4.27).—(A) In manufacturing alkali nitrates by the action of oxides of nitrogen on the corresponding chlorides, the vapours evolved contain about 1% of the oxides of nitrogen and 3% of hydrogen chloride; by treatment with sulphuric acid under pressure the nitrogen oxides are absorbed with the formation of nitrosyl-sulphuric acid, from which they may be recovered by known methods. The acid will absorb up to 5% of its weight of nitrogen as oxide without separation of crystals, and only 0.025% of hydrogen chloride is simultaneously absorbed. (B) An acid solution of alkali chloride saturated at 15° and containing some nitrate is treated under pressure (e.g., 6 atm.) at 35° with a mixture of nitric oxide, oxygen, and nitrogen (e.g., in the volume ratio of 9 : 15 : 76). When absorption of the nitric oxide is complete the pressure is released and solid alkali chloride equivalent to the nitric acid formed is added; on cooling to 15° the corresponding nitrate crystallises out.

A. R. POWELL.

Production of nitrate of lime. APPAREILS ET ÉVAPORATEURS KESTNER (B.P. 279,037, 26.8.27. Fr., 16.10.26).—Coarsely crushed limestone is treated with nitric acid in a tower provided for forced circulation of the carbon dioxide in the direction of the general movement of the materials, thus avoiding the opposition of the gas to the flow of nitric acid and nitrate, a partial vacuum being produced at the outlet of the tower or an increased pressure at its inlet. A heater causes complete disengagement of the carbon dioxide from the newly-prepared calcium nitrate liquor.

W. G. CAREY.

Production of zinc salts [sulphate]. METALLBANK & METALLURGISCHE GES. A.-G. (B.P. 278,747, 7.10.27. Ger., 7.10.26).—Liquors obtained by leaching with dilute sulphuric acid the product resulting from the chloridising roasting of zinciferous pyrites are digested with cement copper to remove dissolved copper as cuprous chloride. The solution is used for leaching a further quantity of roasted material, again denuded of copper, cooled to 5° to allow Glauber's salt to crystallise out, and put through the whole process again to obtain a solution containing 400 g./litre of zinc sulphate. This liquor is freed from iron by treatment with lime and from copper and cobalt by boiling with zinc dust; on cooling to 10° crystals of zinc sulphate free from sodium sulphate are obtained. Alternatively, the purified liquor may be concentrated until it contains 620 g./litre of zinc sulphate and cooled to 60°, whereby sodium sulphate in excess of 70 g./litre crystallises out as the double salt with zinc sulphate. Further cooling to 10° yields pure zinc sulphate. A third method of treating the solution comprises adding sufficient sulphuric acid to provide an excess of 20% over that required to form sodium hydrogen sulphate and evaporating the solution to obtain zinc sulphate monohydrate.

A. R. POWELL.

Manufacture of alumina [from bauxite, clays, etc.]. J. C. SEAILLES (B.P. 277,697, 19.9.27. Fr.,

18.9.26).—To render the alumina content more readily soluble, clays are calcined at 650–750° and bauxite at 500–600°. In either case the calcined product is heated with a solution or suspension in water of an alkaline-earth hydroxide either with or without pressure. With barium hydroxide a solution of barium aluminate is obtained which is separated from the insoluble portion and treated with sodium carbonate or sulphate to obtain a solution of sodium aluminate, from which alumina may be precipitated and sodium carbonate regenerated by known methods; the barium sulphate or carbonate precipitate is converted into hydroxide for further use in the process. An alternative procedure consists in treating the barium aluminate solution with lime to precipitate calcium aluminate (which is converted into the sodium salt as described above), and to regenerate directly barium hydroxide. In case calcium hydroxide is used to decompose the ore the whole of the alumina remains insoluble as calcium aluminate, which is converted into the sodium salt by digestion with sodium carbonate under pressure. The aluminium hydroxide obtained by the above processes is claimed to be practically free from silica and a high recovery is effected.

A. R. POWELL.

Purification of alumina. ALUMINUM CO. OF AMERICA, Assecs. of B. T. HORSFIELD (B.P. 262,405, 3.11.26. U.S., 4.12.25. Addn. to B.P. 248,360; B., 1927, 682).—In the electrothermic treatment of clays and bauxites to obtain a ferrosilicon-titanium alloy and a fluid alumina slag, the charge is so adjusted that the latter contains at least 95% Al_2O_3 . During pouring it is blown with an oxidising blast of air and/or steam and the hollow globules so obtained are treated with an acid solution which will remove all the lime and most of the ferric oxide, titania, and silica. As leach liquor, sulphuric, hydrochloric, or hydrofluoric acid may be used, or mixtures which generate these acids, e.g., sodium chloride and sulphur dioxide in the presence of air. The purified alumina so obtained contains 99.5–99.8% Al_2O_3 .

A. R. POWELL.

Disintegration of liquid alumina. A. L. MOND. From METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 284,131, 7.6.27).—Fused alumina is transformed into small, easily separable crystals suitable for fusion electrolysis by disintegration of the molten mass with rapidly rotating discs or paddles, or by blowing with air or other gases, followed by rapid cooling by water as rain or spray, or by projecting the disintegrated material into a chamber irrigated with water, the particles being collected in a container through which water flows. Means are provided for preventing both premature solidification of the molten alumina and the steam evolved from coming into contact with the hot or non-disintegrated material.

W. G. CAREY.

Production of chromyl chloride. PERMUTIT A.-G. (B.P. 270,711, 3.5.27. Ger., 5.5.26).—An acid chloride, e.g., chlorosulphonic acid, reacts with chromic acid or a chromate in the presence of concentrated sulphuric acid or other diluting agent which does not decompose chromyl chloride; the acid anhydride formed, e.g., sulphur trioxide when chlorosulphonic acid is used, is converted into an acid chloride by the intro-

duction of hydrochloric acid, and then acts on fresh quantities of chromic acid.

W. G. CAREY.

Manufacture of titanium compounds. TITAN CO. A./S. (B.P. 271,085, 11.5.27. Norw., 12.5.26).—The hot solution of titanium and ferrous sulphates obtained by treating ilmenite with sulphuric acid is added slowly with continuous stirring to a quantity of hot mother-liquor from which most of the titanium has been removed by hydrolysis under such conditions that the composition of the solution remains constant while the titanium salt is gradually hydrolysed. As hydrolysis proceeds the liquor containing the suspended precipitate is withdrawn from the precipitation vessel, the precipitate is removed, and the hot mother-liquor returned to the precipitation vessel, or treated separately at increased temperature and pressure in order to precipitate its remaining titanium content.

A. R. POWELL.

Manufacture of a radium preparation. O. HAHN, Assr. to U.S. RADIUM CORP. (U.S.P. 1,655,184, 3.1.28. Appl., 29.7.25. Ger., 15.8.24).—A solution of a lanthanum salt containing a small quantity of a radium salt is treated with hydrofluoric acid to precipitate lanthanum fluoride having radium fluoride dispersed through it. Alternatively, a solution containing radium bromide and thorium nitrate is evaporated to dryness and the residue heated at 500° to give a mixture of radium bromide and thorium oxide. The dispersed radium in such mixtures liberates radon freely.

T. S. WHEELER.

Manufacture of ammonium phosphate. R. GRIESSBACH and K. RÖHRE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,659,198, 14.2.28. Appl., 17.3.26. Ger., 23.3.25).—See B. P. 255,766; B., 1926, 821.

Roasting of gas-purifying substances (B.P. 271,854). **Sulphuric acid from acid tar** (B.P. 277,619 and 281,547). **Ammonia saturator** (U.S.P. 1,654,159).—See II. **Alkali arsenates** (U.S.P. 1,654,527–8).—See X.

VIII.—GLASS; CERAMICS.

Effectiveness of eye protection glasses and its characterisation. L. BLOCH (Gas- u. Wasserfach, 1927, 70, 1229–1232).—The penetrability of eye-protection glasses is most easily determined for the visible rays, by measuring with a photometer the intensity of a source of light before and after interposing the given glass. The value usually recorded is the “dimming index,” i.e., the negative logarithm of the penetrability. Instead of taking the visible spectrum as a whole, separate observations may be made with red, green, and blue rays by placing a red, green, or blue glass in front of the eyepiece of the photometer. The dimming indices for the three colours afford a means of characterising the colour of the given glass. Similar measurements for infra-red rays are easily made by determining the diminution caused by interposing the given glass between an electrically-heated glowing wire and a thermopile. For measurements with ultra-violet rays the light from a quartz lamp is passed through a screen (e.g., “black glass” of the Sendlinger Optischer Glaswerke) which allows only the ultra-violet rays to pass. When these rays fall on to white drawing paper the latter shines out

brightly, and if a test glass is interposed its dimming effect may be measured. The dimming indices of different coloured glasses are less widely different for infra-red rays than for visible rays, whilst the indices for ultra-violet rays bear no close relation to those for visible and infra-red rays.

W. T. K. BRAUNHOLTZ.

PATENTS.

Manufacture of acid metal phosphate [glass].

H. BLUMENBERG, JUN., Assr. to STOCKHOLDERS' SYND. (U.S.P. 1,654,404, 27.12.27. Appl., 23.12.24).—A metallic compound, *e.g.*, lead oxide or potassium carbonate, is heated at 400–700° with phosphoric acid (4–14 mols.) to yield a glass of value in the manufacture of enamels with low m.p.

T. S. WHEELER.

Manufacture of ceramic material. G. KNUDSEN (B.P. 260,298, 23.10.26. Norw., 24.10.25).—Shaped ceramic or refractory products are made from a mixture of talc and raw or burnt magnesite mixed in such proportions as to obtain magnesium orthosilicate after firing. The materials are finely ground, mixed with a binding agent such as tar or molasses to obtain a suitable degree of plasticity and with a filler such as olivine or artificial magnesium orthosilicate from a previous charge, formed into shape, and fired at 1000–1400° for 48 hrs. or until the greater part of the charge is converted into Mg_2SiO_4 . A. R. POWELL.

Manufacture of heat-insulating material. W. W. ODELL (U.S.P. 1,657,582, 31.1.28. Appl., 23.4.25).—Plastic clay and prepared alkaline peat containing only sufficient water to give plasticity for working and moulding are intimately mixed and macerated, the mixture then being moulded, dried, and burnt.

W. G. CAREY.

Refractory material. V. M. GOLDSCHMIDT and R. KNUDSEN (B.P. 283,791, 22.6.27).—A furnace or other apparatus which is exposed to high temperatures and chemical action is made from natural olivine rock containing less than 10% FeO, and/or from blocks made of olivine fragments rammed and heated with or without a binding agent, *e.g.*, colloidal magnesium silicate, magnesium oxide, tar, pitch, etc.

W. G. CAREY.

Production of vitreous silica. BRIT. THOMSON-HOUSTON CO., LTD., Asses. of L. B. MILLER (B.P. 282,733, 5.12.27. U.S., 29.12.26).—See U.S.P. 1,628,468; B., 1927, 523.

Production of highly refractory magnesite masses. F. BAUMHAUER, Assr. to DYNAMIDON-WERK ENGELHORN & Co., G.M.B.H. (U.S.P. 1,659,476, 14.2.28. Appl., 16.2.24. Ger., 20.2.23).—See B.P. 211,873; B., 1925, 243.

IX.—BUILDING MATERIALS.

Flow of heat through limestone and lime. R. T. HASLAM and V. C. SMITH (Ind. Eng. Chem., 1928, 20, 170–174).—The mathematical treatment of the rate of heat flow through solids is developed for the case of limestone (in which dissociation occurs), but only on the assumption of the limestone being a "semi-infinite block." The rate of heat transference

through slabs of moderate size was determined experimentally by means of embedded thermocouples, the sides of the slabs being insulated so that heat transference was in one direction only. The black-body coefficients were determined as about 0.5 using temperatures below that of the dissociation of limestone. With a furnace temperature of 1060° the ratio between the time required for the dissociation temperature to penetrate a given distance and the calculated time (ignoring the thermal requirements of dissociation) was found to be 0.425. This experimental figure was then used for the calculation of the time required for the complete dissociation of spheres of limestone of given diameter at given temperatures. The (linear) relationship between the radius of such a sphere and the square root of the time required is depicted in a series of curves. C. IRWIN.

Laboratory tests on physical properties of water-bearing materials. N. D. STEARNS (U.S. Geol. Survey, Water-Supply Paper 596r, 1927, 121–176).—Methods of sampling water-bearing strata and of determining their apparent sp. gr., mechanical composition, porosity, moisture equivalent, and permeability to water are described in detail with reference to certain American water-bearing gravels, tables showing the physical properties of which are included.

A. R. POWELL.

Principles of kiln-seasoning of timber. I. Types of commercial kilns in use. S. T. C. STILLWELL (Dept. Sci. Ind. Res., Forest Prods. Res., Spec. Rept. No. 2, 1928, 11 pp.).

PATENTS.

Impregnation of wood with two or more liquids.

A. DESSEMOND (B.P. 283,703, 1.12.26).—The wood is subjected to the action of a vacuum after each impregnation and the liquid extracted is measured so that the degree and duration of the vacuum are controlled, and the quantity of impregnating liquid in the wood is proportioned. Suitable apparatus is described.

W. G. CAREY.

Composition for impregnation of wood. B. WURZSCHMITT, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,654,229, 27.12.27. Appl., 17.6.26. Holl., 15.6.25).—The process of U.S.P. 1,500,066 (B., 1924, 749) is modified in that formaldehyde is added to the solutions therein described to inhibit their corrosive action on iron.

T. S. WHEELER.

Varnishing of wood. J. PAISSEAU (Addn. No. 31,459, 3.8.25, to F.P. 613,502).—Nitrocellulose lacquers adhere better to wood which has previously been treated with nitric or mixed acid, washed, and dried.

C. HOLLINS.

Protection of wood. P. BARTSCH (Re-issue 16,880, 14.2.28, of U.S.P. 1,374,806, 12.4.21).—See B., 1921, 434 A.

Bituminous composition (U.S.P. 1,659,554).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Heat of the A2 and A3 transformations in carbon steels. S. UMINO (Sci. Rep. Tôhoku Imp. Univ., 1927, 16, 1009–1030).—From determinations of the heat

content and true sp. heat at high temperatures of steels containing 0.04–0.077% C the heat of transformation of iron at the A2 point is deduced as 3.63 g.-cal./g. and at the A3 point, 5.35 g.-cal./g. The A2 transformation in pure iron takes place over a range of 130–140°, and the A3 transformation requires an appreciable time to become complete at 930°. Both heats of transformation are decreased by the presence of carbon, being 3.23 and 3.50 g.-cal./g. respectively for the 0.35% C steel, and 3.18 and 0.8 g.-cal./g. for the 0.77% C steel. The heat of the A1 transformation increases rapidly with the carbon content from 0.67 g.-cal./g. with 0.04% C to 13.6 g.-cal./g. with 0.77% C. A. R. POWELL.

Tempering changes in carbon steels. R. HAY and R. HIGGINS (J. Roy. Tech. Coll., Glasgow, 1927, [4], 62–76).—The tempering changes which take place in a 0.26% carbon steel have been investigated by determining the Brinell hardness, specific volume, yield stress, maximum stress, percentage elongation, and Charpy impact value of specimens which had been water-quenched from 1000° and then tempered from laboratory temperatures to 650°. Pronounced maxima or minima occur in the property-temperature curve at about 100°, 250°, and 350°. Brinell hardness and specific volume determinations carried out on steel quenched at temperatures from 400° to 1150° show that an appreciable amount of iron carbide goes into solution below 650°. When such steels are kept for two months, those specimens quenched below 650° undergo a considerable increase in hardness and specific volume, probably owing to deposition of finely dispersed carbide through the α -iron. Tempering at 50° brings about a fall in hardness, which is ascribed to coagulation of this carbide. The change at 100° is thought to be due to decomposition of iron carbide in α -iron, whereas the change at 250° is attributed to the decomposition of residual austenite in the quenched specimens. The change at 350° is accounted for by the assumption that all the remaining austenite is not transformed immediately to martensite and then to troostite, but that the change is periodic and takes a certain time to go to completion. L. M. CLARK.

Action of water, air, oxygen, and carbon dioxide on the corrosion of iron. K. INAMURA (Sci. Rep. Tôhoku Imp. Univ., 1927, 16, 979–986).—Pure water free from dissolved gases has only a very slight rusting action on iron which is barely influenced by the addition of pure sodium chloride. Passage of pure oxygen through either the water or the salt solution causes a rapid increase in the rate of corrosion. Carbon dioxide alone increases the rate of corrosion, but not nearly to the same extent as oxygen alone; it, however, accelerates the corrosion due to oxygen, but apparently to a smaller degree than is generally accepted. A. R. POWELL.

Investigation of the corrosion of metals [iron] with a thermobalance. K. INAMURA (Sci. Rep. Tôhoku Imp. Univ., 1927, 16, 987–997).—The rate of corrosion of a sample of commercial iron in various salt and acid solutions has been followed by means of the Honda thermobalance (*ibid.*, 1915, 4, 97). In solutions of sodium carbonate and sodium hydrogen carbonate, the specimen gained in weight linearly with the time of immersion owing to the slow formation of

ferrous hydroxide over the surface; in dilute acid solutions the decrease in weight was also a linear function of the time, but, owing to adhesion of hydrogen bubbles, observation in very dilute acids (0.0025*N*) was uncertain. In all the solutions tested, corrosion started at several spots and slowly spread from these over the surface of the metal. A. R. POWELL.

Manganese in steel and pig iron. Volumetric determination by the vanadate method. L. E. STOUT and G. C. WHITAKER (Ind. Eng. Chem., 1928, 20, 210–212).—The method proposed is the inverse of the determination of vanadium in steel by oxidation with permanganate. Standard vanadyl sulphate solution is prepared by dissolving ammonium metavanadate in dilute sulphuric acid and reducing with sulphurous acid in excess. It is standardised on steel of known manganese content and is very stable. A 0.2 g. sample of steel is dissolved in 20 c.c. of 1:3 sulphuric acid, and 5 c.c. of 10% ammonium persulphate solution are added. After cooling, 10 c.c. of silver sulphate solution are added, the mixture is shaken, and 10 c.c. of ammonium persulphate are added. After 30 min., 10 c.c. of sodium chloride solution are added. The two solutions are equivalent so that the silver ion is quantitatively removed. The solution is then titrated with vanadyl sulphate solution. The first oxidation converts iron into ferric sulphate. The second oxidation in presence of silver sulphate converts manganese into permanganic acid. If pig iron is to be tested, the only variation is in removing undissolved graphite before adding silver sulphate. A large number of results of good accuracy are tabulated, and details as to the time required for complete oxidation with varying quantities of silver sulphate are given. C. IRWIN.

Determination of minute amounts of cobalt in steel. W. J. AGNEW (Analyst, 1928, 53, 31–32).—1 g. of steel is dissolved in aqua regia, the solution evaporated to dryness, the residue ignited, dissolved in hydrochloric acid, the solution evaporated to a small bulk, slightly diluted, iron (or chromium) separated by an emulsion of zinc oxide, the volume made up to 200 c.c., and 100 c.c. (0.5 g. of steel) are filtered off. To the filtrate 5 c.c. of concentrated hydrochloric acid are added, hydrogen sulphide is passed through to remove copper, and, after filtration, the filtrate is boiled and 0.2 g. of α -nitroso- β -naphthol dissolved in 10 c.c. of strong acetic acid is added. After boiling and allowing to settle for $\frac{1}{2}$ hr. the precipitate is collected on a filter, washed with 5% hydrochloric acid and hot water, ignited at about 800°, the residue dissolved in about 10 drops of hot concentrated hydrochloric acid, and the diluted solution placed in a Nessler tube. The colour produced by adding 10 c.c. of 1:1 ammonia solution and 5 c.c. of 0.1% potassium ferricyanide solution is matched by adding a cobalt solution (containing 0.001 g. Co per c.c.) to 10 c.c. of ammonia, 10 drops of hydrochloric acid, and 5 c.c. of the ferricyanide solution. If more than 2 mg. or 0.4% Co is present, a gravimetric method is more suitable. The sensitiveness of the test is 1 part in 5×10^5 . D. G. HEWER.

Corrosion of copper and brass. K. INAMURA (Sci. Rep. Tôhoku Imp. Univ., 1927, 16, 999–1008).—

During a period of five weeks the rate of corrosion of copper is a linear function of the time of immersion in *N*-ammonium chloride, 0.01*N*-hydrochloric acid, 0.01*N*-nitric acid, 0.01*N*-sulphuric acid, and distilled water, the rate decreasing in the above order. In 0.01*N*-solutions of ammonia, sodium chloride, ammonium chloride, sodium carbonate, and potassium hydroxide the daily loss in weight decreases slowly at first, then more and more rapidly until finally corrosion ceases. The behaviour of 70 : 30 brass to the first of the above series of reagents is similar to that of copper, but in the case of the second series the loss in weight-time curves are straight lines, corrosion proceeding at a steady rate over the whole period.

A. R. POWELL.

Colorimetric determination of small amounts of iron in zinc. W. J. ANGEW (Analyst, 1928, 53, 30).—Zinc (2 g.) is dissolved in 20 c.c. of hydrochloric acid, a few c.c. of nitric acid are added to oxidise any iron, the solution is treated with 0.01 g. of uranium (as uranyl nitrate solution) to agglomerate the iron, ammonia solution is added in slight excess, the liquid boiled, and, after 20 min., the precipitated ferric and uranium hydroxides are collected on a filter and dissolved in 10 c.c. of 1 : 1 hydrochloric acid. The solution is then placed in a Nessler tube, made almost alkaline with ammonia solution, and 1 c.c. of a 10% aqueous solution of sodium salicylate added, followed by ammonia in slight excess, as seen by the yellow colour due to uranium. Finally, it is acidified with acetic acid until the purple colour, due to iron, is developed, and the uranium yellow is destroyed. After addition of 10 c.c. of 1 : 1 acetic acid the colour is compared with that of a solution made by adding a standard iron solution (made from ferric sulphate and containing 0.0001 g. of iron per c.c.) to a Nessler glass containing the same quantity of uranium solution of the same acidity. The sensitiveness of the test is 1 pt. in 2.5×10^6 , and the purple colour is stable for 48 hrs. in the presence of acetic acid, but is destroyed by sulphuric, hydrochloric, and nitric acids.

D. G. HEWER.

PATENTS.

Production of soft workable grey iron. L. MELLERSH-JACKSON. From MASCHINENFABRIK ESSLINGEN (B.P. 260,619, 29.10.26).—The smelted iron is superheated at 1400° or more, the superheating temperature increasing with the content of carbon and silicon. The product is afterwards cast at the normal casting temperature. The wall-thickness of the castings and the composition of the iron with regard to its carbon and silicon content must be taken into account when selecting the superheating temperature.

M. E. NOTTAGE.

Heat-treatment of metals [hardening of steel]. C. L. IPSEN, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,658,682, 7.2.28. Appl., 10.7.25).—An empty furnace is heated to a definite quenching temperature and a charge temperature indicator is set to a definite reference mark at this temperature. The charge is introduced, heated, and quenched when the indicator reaches approximately the reference mark.

J. S. G. THOMAS.

Preventing the dissolution of iron and steel in sulphuric acid. NEWPORT Co. (B.P. 259,200, 20.9.26.

U.S., 30.9.25).—The dissolution of iron and steel in sulphuric acid of strength less than 90% may be prevented or retarded by the addition of 1% or less of thiourea or its substitution products.

M. E. NOTTAGE.

Alloy steel. W. R. SHIMER and R. H. CHRIST, Assrs. to BETHLEHEM STEEL Co. (U.S.P. 1,659,055, 14.2.28. Appl., 5.5.25).—An alloy containing up to 2% Cr, up to 2% W, and not more than 0.3% C is claimed.

F. G. CROSSE.

Nickel-cobalt-iron alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 282,901, 1.10.26).—An alloy, having a high magnetic permeability in a minute field of force and also a high magnetic induction in a moderately strong field, is composed of 55–65% Ni, 20–30% Co, and 15–25% Fe, with or without 1–5% of a fourth element, *e.g.*, chromium, which increases the electrical resistance; a small quantity of a deoxidiser such as magnesium may also be added.

M. E. NOTTAGE.

Hydrometallurgical process. G. H. BUCHANAN, G. B. WINNER, and E. L. TUCKER, Assrs. to AMER. CYANAMID Co. (U.S.P. 1,654,918, 3.1.28. Appl., 16.11.23).—Ore containing copper, silver, and gold is cyanided, and the solution is treated with zinc to precipitate silver and gold. The residual solution is acidified to separate copper cyanide, which is treated with sodium chloride, sulphuric acid, and iron to form copper and hydrogen cyanide.

T. S. WHEELER.

Copper-extraction process. W. E. GREENAWALT (U.S.P. 1,654,930, 3.1.28. Appl., 13.8.26).—The process of U.S.P. 1,483,056 (B., 1924, 340) is modified in that the solution to be electrolysed is treated with hydrogen sulphide to decompose any sulphur dioxide present.

T. S. WHEELER.

Aluminium alloys. T. GOLDSCHMIDT A.-G. (B.P. 260,275, 20.10.26. Ger., 21.10.25).—The mechanical and physical properties of aluminium alloys are improved by heating the alloy to redness and quenching, followed by a sequence of ageing treatments at atmospheric and higher temperatures. Alloys containing 88–98% Al may possess tensile strengths of 36–55 kg./mm.² and elongations of 18–26%.

C. A. KING.

Aluminium alloys. J. STONE & Co., LTD., and H. J. MAYBREY (B.P. 283,760, 26.2.27).—The structure of aluminium alloys, particularly those containing silicon, is improved by treating the alloy with boron exothermically. A suitable reaction mixture contains a compound of boron, powdered aluminium, and oxygen carrier, *e.g.*, potassium chlorate, barium peroxide, or manganese dioxide, if the final presence of manganese is desired.

C. A. KING.

Manufacture of aluminium-nickel-silver alloys. R. K. HEZLET and R. GENDERS (B.P. 283,994, 16.10.26).—In a “nickel-silver” alloy the combined content of nickel and aluminium is more than 15%, up to 2.5% Al being present. A particular composition consists of 20% Ni, 50% Cu, 0.2–0.3% Al, and the remainder zinc. When casting the alloy the cross-sectional dimensions of the molten stream should be approximately the same as that of the mould.

C. A. KING.

Metal compositions [aluminium alloys]. K. BERNHOEFT, Assr. to GEN. ELECTRIC Co. (U.S.P.

1,658,702 and 1,658,757, 7.2.28. Appl., [A] 19.8.26, [B] 7.1.27. Ger., [A] 17.12.25, [B] 19.3.26).—(A) An alloy of 98.4% Al and 1.6% Cd. (B) Aluminium is alloyed with 0.1–1% Cu and 0.5–3% Cd. F. G. CROSSE.

Alteration of the colour of the magnesium flame. BOEHM-WERKE A.-G. (G.P. 445,181, 1.10.25).—Magnesium foil or ribbon is coated with salts which impart the desired colour to the flame when it is burnt.

L. A. COLES.

Production of metallic antimony, alloys of antimony, and alkali arsenates. H. M. BURKEY, Assr. to AMER. METAL CO., LTD. (U.S.P. 1,654,527—8, 3.1.28. Appl., [A] 11.1.26, [B] 27.10.26).—(A) A mixture of antimony trioxide, arsenic trioxide, and sodium carbonate is fused to yield antimony, sodium arsenate, and carbon dioxide. By treating the fused mixture with air a relatively small quantity of antimony trioxide can be used. (B) The process is performed in presence of molten lead to alloy the antimony formed.

T. S. WHEELER.

Removing, purifying, and recovering oil from tin plate. Separating oil from aqueous emulsions. G. B. ELLIS. From AMER. SHEET & TIN PLATE CO. (B.P. 283,614 and 283,830, 13.9.26).—(A) In an apparatus for cleansing tinned plates and recovering palm oil therefrom, the plates, after passing through the tinning and oiling baths, are passed through a dilute, hot, alkaline, aqueous solution prior to the final cleansing and polishing with bran. By this treatment the oil is also freed from flux (zinc chloride) and thus rendered fit for further use. (B) The emulsion is destroyed by acidification or mechanical treatment (centrifuging), and the oil is separated and recovered in a special apparatus described.

C. O. HARVEY.

Treatment of manganese ores. S. G. S. DICKER. From J. C. WIARDA & Co. (B.P. 284,098, 1.3.27).—Manganese ore (carbonate or dioxide) is ground to 200-mesh and heated at 600° for several hours in a reducing atmosphere or with a reducing agent, whereby the manganese content of the ore is converted into manganous oxide. The hot material is discharged directly into a hot 15% solution of ammonium sulphate or chloride, which dissolves the manganous oxide with evolution of ammonia. The mixture is digested at 80° with agitation until all the ammonium salt is decomposed and the liberated ammonia expelled from the solution. Manganous hydroxide is precipitated from the filtered solution by treatment with an aqueous solution of the ammonia expelled in the first stage, thereby regenerating ammonium sulphate for a subsequent leaching operation. The precipitate is collected, washed, dried, and dehydrated to manganous oxide by heating at 825°; air is excluded throughout the treatment.

A. R. POWELL.

Roasting of vanadiferous ores and compounds. H. T. KOENIG (U.S.P. 1,654,820, 3.1.28. Appl., 13.10.25).—The ore is heated with sodium chloride and sulphuric acid at 600–900° to liberate free vanadic acid.

T. S. WHEELER.

Desulphidising and desulphurising of sulphur minerals. M. F. COOLBAUGH and J. B. READ, Assrs. to COMPLEX ORES RECOVERIES Co. (U.S.P. 1,657,711, 31.1.28. Appl., 4.8.22).—In a multi-stage roasting

operation sulphide minerals are heated in an atmosphere containing an excess of sulphur trioxide to form sulphates, which are then rapidly dissociated by withdrawing the partial pressure of sulphur trioxide. C. A. KING.

Production of rock wool. E. R. POWELL (U.S.P. 1,656,828, 17.1.28. Appl., 5.1.27).—Suitable raw material is melted in the presence of hot gases from an oxidising flame before being run off and converted into the form of wool. M. E. NOTTAGE.

Deflocculation of colloids. W. O. BORCHERT. Assr. to NEW JERSEY ZINC CO. (U.S.P. 1,655,045, 3.1.28. Appl., 1.4.25).—Runaway extract, a waste product of tanneries, is used to deflocculate the colloidal matter present in mineral pulps prior to froth flotation.

T. S. WHEELER.

Manufacture of [metallic] catalysts. I. G. FAR-ENIND. A.-G. (B.P. 281,218, 5.5.27. Ger., 27.11.26).—Catalysts consisting of metals such as nickel, cobalt, or copper, or mixtures of these, may be made from aqueous solutions of their salts by means of a more electro-positive metal such as iron, zinc, or aluminium in the presence or absence of a non-metallic carrier such as kieselguhr, silica gel, or carbon. The metal may be separated in a neutral, acid, or alkaline solution; in an alkaline solution the oxide of the electro-positive metal is simultaneously produced and itself activates the catalyst. M. E. NOTTAGE.

Rust-proofing process. W. H. ALLEN (U.S.P. 1,654,716, 3.1.28. Appl., 7.5.26).—The process of B.P. 8667 of 1906 (B., 1907, 207) is modified in that the surface of the article under treatment is rendered smooth by rubbing it with cotton fabric during immersion in the rust-proofing solution. T. S. WHEELER.

Disintegration of metals. E. J. HALL, Assr. to METALS DISINTEGRATING CO., INC. (U.S.P. 1,659,291, 14.2.28. Appl., 20.12.17. Renewed 30.10.22).—See B.P. 121,600; B., 1919, 826.

Zirconium alloy. G. R. FONDA, Assr. to GENERAL ELECTRIC Co. (U.S.P. 1,658,712, 7.2.28. Appl., 30.8.24).—See B.P. 239,235; B., 1925, 908.

Washing of ores (B.P. 266,723).—See II. Cleaning of metal surfaces (U.S.P. 1,658,222).—See XI.

XI.—ELECTROTECHNICS.

Electrolytic precipitation of latices. SCHOLZ.—See XIV.

PATENTS.

Electric furnace. C. A. CADWELL, Assr. to ELECTRIC RAILWAY IMPROVEMENT Co. (U.S.P. 1,657,785, 31.1.28. Appl., 29.4.20).—Electrodes extend into a chamber, and a resistor providing paths of varying resistance for passage of current from one electrode to the other is arranged in the chamber between the electrodes.

J. S. G. THOMAS.

Electric furnace. J. L. BERNARD (U.S.P. 1,658,071—2, 7.2.28. Appl., [A, B] 15.12.26).—(A) A wheel-supported furnace body runs on tracks on a tilting base from which a frame carrying a vertically adjustable roof extends upwards. Means are provided for adjusting the roof and for locking the furnace body in the frame. (B) In a furnace constructed in accordance with (A) jacks are

attached to the tilting base for raising the furnace body into contact with the roof, and means are provided on the frame for guiding the furnace body.

J. S. G. THOMAS.

Electro-cleaning [of metal surfaces]. R. M. BURNS and C. W. WARNER, Assrs. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,658,222, 7.2.28. Appl., 10.2.25).—Electric current is passed between suitable cathodes and metallic surfaces to be cleaned, which are immersed in a solution of phosphoric acid maintained at 50° and at 70% concentration.

J. S. G. THOMAS.

Construction of vacuum tubes. H. E. METCALF, Assr. to MAGNAVON Co. (U.S.P. 1,657,636, 31.1.28. Appl., 23.7.24).—One of the electrodes of a vacuum tube is of German silver, and is heated to throw off the copper and zinc and deposit them on the walls of the tube, the nickel of the alloy being left in place to form an electrode.

W. G. CAREY.

XII.—FATS; OILS; WAXES.

Examination of residues from the refining of oils and fats. J. DE TOURNADRE (Chim. et Ind., 1928, 19, 39—40).—The process of oil refining includes the removal of free fatty acids, the bleaching of the neutral oil, and the removal by steam distillation of substances giving an offensive odour or taste. The chief by-product is obtained in the first stage, and is either a free fatty acid (from decomposition of a lime soap) or a soda soap containing a large quantity of neutral oils. These substances are sold to the soapmaker on the basis of their fatty acid content. The free acid is determined by difference from a determination of moisture and of residue insoluble in carbon disulphide. In the case of the soda soap a given quantity is decomposed with strong hydrochloric acid. If a clear separation is obtained wax or stearic acid is added if necessary to bring the acid to the solid state, when it is separated. If separation is not good the whole is dissolved in hot water, cooled, and the fatty acid extracted with carbon disulphide.

C. IRWIN.

Halphen reaction for cottonseed oil as a general reaction for oils of the families *Malvaceæ*, *Tiliaceæ*, and *Bombacaceæ*. S. IVANOV (Ber. Deut. bot. Ges., 1927, 45, 588—591).—The substance causing the Halphen test colour in cottonseed oil is located in the seed, and is extracted with the oil. It is not present in the petals or colouring matter. The reaction is specific for the order *Columnifera*, including *Malvaceæ*, *Tiliaceæ*, and *Bombacaceæ*, but not *Sterculiaceæ*.

A. G. POLLARD.

Constitution of elæostearic acid. F. FRITZ (Farben-Ztg., 1928, 33, 1224—1225).—A brief account of the work of various investigators on the constitution of elæostearic acid as revealed by its oxidation products. The validity of Böeseken and Ravenswaay's formula for the acid (cf. B., 1925, 813) is considered to be upheld.

S. S. WOOLF.

Determination of oleic and linoleic acids in an oil; determination of the bromine index. Y. VOLMAR and B. SAMPAHL (J. Pharm. Chim., 1928, [viii], 7, 106—109).—For the determination of oleic and linoleic acids in oils containing no other unsaturated

acids, calculation of the percentage amounts from the iodine value of the mixture gives very discordant results compared with those given by the gravimetric tetrabromide method of Eibner and Muggenthaler for the determination of linoleic acid. The following volumetric method gives results comparable with those of the above gravimetric method. The unsaturated acids (0.5 g.) in 15 c.c. of dry ether are cooled to -10° , and bromine is added drop by drop until the brown colour persists. After keeping at -10° for 2 hrs. the excess of bromine is removed with 5% aqueous sodium thiosulphate solution, the ether evaporated, and the residue dissolved in 100 c.c. of 95% alcohol, 20 c.c. of the solution are boiled for 5 hrs. with 5 c.c. of 60% aqueous potassium hydroxide solution, and, after cooling, the bromine is determined by Volhard's method. E. H. SHARPLES.

Oil bromide films and their use in determining the halogen absorption of oils. II. TOMS (Analyst, 1928, 53, 69—77).—The "insoluble bromide" obtained from most drying oils has been debrominated with difficulty and the presence of hexabromostearic acid confirmed, whilst the presence of tetrabromostearic acid has been rendered more probable by the isolation of a thick oil, apparently a liquid form of this acid. A satisfactory micro-method for determining the unsaturation value of non-volatile oils consists in spreading a thin film of the oil in a single drop, about 0.2 mm. thick, on a weighed microscope slide, and placing the slide in a wide tube closed at each end with a waxed cork and containing a boat with a few drops of bromine. After 20—30 min. the slide is withdrawn, excess of bromine removed, and the slide again weighed; the bromine value is calculated from the increase in weight. This value multiplied by the at. wt. of iodine and divided by the at. wt. of bromine gives the ordinary iodine value, and agrees well with theory except in the peculiar case of tung oil, the appearance of the brominated films of which is characteristic. A method is given for calculating the percentage of α -elæostearic triglyceride in tung oil assuming the presence in the oil of two types of substances, viz., (a) those containing non-conjugated systems of double linkings and giving the same iodine value regardless of the method used, and (b) the glyceride of α -elæostearic acid which gives only two thirds its theoretical value with Wijs' solution in 2 hrs., but its full value by the bromide vapour method.

D. G. HEWER.

Drying of oils. EIBNER.—See XIII. **Determination of mustard oil.** SACCARDI and PRIERI.—See XIX.

PATENTS.

Detergent, cleansing, and polishing compositions. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, and E. CHAPMAN (B.P. 284,367, 21.9.26).—A cleansing composition for tiles, walls, etc. comprises a sulphonated (or sulphonated and alkylated) high-boiling mineral oil fraction in powder or paste form or in solution together with abrasive material (whiting, pumice), with or without addition of thickening and tinting agents, an organic solvent, soap, or a sulphonated oil. The first-mentioned ingredient increases the cleansing efficiency by ensuring rapid and complete contact with the surface to be cleansed.

C. HOLLINS.

Manufacture of cleansing and emulsifying agents.

I. G. FARBENIND. A.-G. (B.P. 260,243 and Addn. B.P. 283,786, 13.10.26. Ger., [B] 21.10.25).—(A) The oily products practically insoluble in water and consisting chiefly of higher aliphatic alcohols, obtained by catalytic hydrogenation of oxides of carbon or by cracking operations employing sulphuric acid, are mixed with a soap and an aliphatic alcohol of low b.p., and, if desired, an aliphatic, aromatic, or hydroaromatic hydrocarbon. (B) Soft soap, grain soap, or other soap-like substance is used as the "soap" constituent of the mixture.

S. S. WOOLF.

Digesters for use in the whale oil industry.

T. ARENTZ (B.P. 271,111, 14.5.27. Norw., 15.5.26).

Waterproofing composition (U.S.P. 1,658,540).—

See II. Recovery of oil from tin plate (B.P. 283,614 and 283,830).—See X. Resins from cashew nut-shell oil (B.P. 283,803).—See XIII.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Negative catalysis as a means of improving the drying of oils. A. EIBNER (Farben.-Ztg., 1928, 33, 1222—1224).—A general discussion of the functions of negative catalysts, with special reference to the effects of undercoats on coats subsequently applied and the neutralisation of such effects by "flatting" the undercoats.

S. S. WOOLF.

Protective paint from rubber. H. GRAY (Ind. Eng. Chem., 1928, 20, 156—158; cf. Fisher, B., 1928, 61).—Films of thermoprene on metal exhibit good adhesion and low permeability and exert a marked protective action against corrosion; they are tough and free from brittleness at 0°.

D. F. TWISS.

Arsenic in coated papers etc. STERN.—See V. **Pigments and rubber.** WINKELMANN and CROAKMAN.—See XIV.

PATENTS.

Protective compositions particularly for anti-corrosive purposes. A. A. VAN DER MEULEN (B.P. 283,664, 28.10.26).—A mixture of 1—2 pts. of phenol and 40 pts. of linseed oil is claimed.

S. S. WOOLF.

Fluorescent paint. L. J. BUTTOLPH, Assr. to COOPER HEWITT ELECTRIC CO. (U.S.P. 1,658,476, 7.2.28. Appl., 17.12.24).—A mixture of benzene, anthracene, rubber, and a vulcaniser is used.

F. G. CROSSE.

Production of [pigment] mixtures containing metal powder. M. RAGG and F. RAHTJEN (B.P. 284,172, 1.10.27).—Mixtures containing metal powder are prepared by atomising the molten metal or alloy and projecting it against the remaining ingredients of the mixture, e.g., pigments, fillers, in suitable apparatus.

S. S. WOOLF.

Plastic and lacquer compositions of nitrocellulose. CANADIAN ELECTRO PRODUCTS CO., LTD., Assees. of H. W. MATHESON (B.P. 270,650, 9.3.27. U.S., 4.5.26).—Esters of mandelic acid are used as plasticisers for plastic and lacquer compositions of nitrocellulose.

S. S. WOOLF.

Compositions of matter containing esters or ethers of carbohydrates. J. SCHINDELMEISER (B.P. 283,619, 6.10.26).—The use of esters of borneol, iso-

borneol, and terpineol in paint or varnish compositions containing esters or ethers of cellulose or other similar carbohydrates is claimed.

S. S. WOOLF.

Manufacture of solutions of organic compounds.

I. G. FARBENIND. A.-G. (B.P. 256,229, 26.7.26. Ger., 1.8.25).—Dialkyl ethers of ethylene, propylene, or butylene glycol are used as solvents for natural or artificial resins, e.g., in cellulose lacquers.

S. S. WOOLF.

Reaction products of cashew nut-shell oil. M. T. HARVEY (B.P. 283,803, 11.10.26).—Cashew nut-shell oil is condensed with aldehydes, in the presence or absence of catalysts, to produce synthetic resins that may subsequently be hardened.

S. S. WOOLF.

Manufacture of condensation products of carbamide and formaldehyde. I. G. FARBENIND. A.-G. (B.P. 260,253, 16.10.26. Ger., 21.10.25).—Lacquers are prepared directly by mixing solutions of carbamide and paraformaldehyde in an organic solvent, water being excluded as far as possible, and incorporating natural or artificial resins, cellulose esters, and colouring matters before or during the carbamide-aldehyde condensation.

S. S. WOOLF.

Manufacture of a condensation product. P. W. GRIFFITH, Assr. to AMER. CYANAMID CO. (U.S.P. 1,658,597, 7.2.28. Appl., 16.8.24).—A product is formed by the interaction of guanidine, carbamide, and formaldehyde.

B. FULLMAN.

Varnishing of wood (Addn. F.P. 31,459).—See IX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Absorption of hydrogen under high pressure by rubber, and behaviour of rubber after release of pressure. G. TAMMANN and K. BOCHOW (Z. anorg. Chem., 1928, 168, 322—324).—If rubber and zinc are placed in 2*N*-sulphuric acid, and the whole is subjected to a pressure of 550 or 1150 kg./cm.², the rubber takes up a considerable amount of hydrogen. If now the pressure is released, the rubber swells considerably, numerous small bubbles appearing in it, but after 15—20 min. about 0.8% of the hydrogen has escaped. After this point, the amount of hydrogen evolved is proportional to the pressure at which the rubber was saturated with the gas, so that the absorption coefficient must be practically independent of the pressure, but transparent rubber and black unvulcanised rubber give up their hydrogen more rapidly than do the red and grey varieties.

R. CUTHILL.

Effect of certain metallic salts on the ageing of a [rubber] "tread compound." B. S. TAYLOR and W. N. JONES (Ind. Eng. Chem., 1928, 20, 132—133).—Ageing experiments with an otherwise typical black "tread compound" by the Geer oven and Bierer-Davis bomb methods show that the presence of as little as 0.1% of copper oleate or manganese oleate is distinctly detrimental. Ferric stearate was less deleterious, but 0.5% had a distinct adverse effect. Mercuric stearate between 0.1 and 1% was relatively innocuous. The bomb test at 70% was much more severe than the oven test at the same temperature. Application of cupric chloride to the surface of the test strips has a similar effect to the incorporation of cupric stearate.

D. F. TWISS.

Effect of the proportion of sulphur in vulcanising [rubber] reclaim. R. E. CARLIDGE and H. L. SNYDER (Ind. Eng. Chem., 1928, 20, 137—140).—Comparison is made of the tensile characteristics and of the proportion of additional combined sulphur in vulcanised products obtained from an alkali "tyre reclaim," when vulcanised with various percentages of sulphur (from 1½ to 6%) or for different periods. The best physical properties with the simple mixture of reclaim and sulphur are obtained with 3% S; in a mixture of equal proportions of the reclaim and new rubber together with carbon black and other typical ingredients, and allowing 4% of sulphur for the vulcanisation of the new rubber, the best proportion for sulphur to reclaim is 2—2½%.

D. F. TWISS.

Hardness tester for rubber. E. C. ZIMMERMAN and R. W. BROWN (Ind. Eng. Chem., 1928, 20, 216—218).—A "penetrometer" is described in which the alteration in stress between minimum and maximum penetration is made negligibly small by the use of a long spring. In order to render the penetrometer applicable over a wide range of hardness, three springs corresponding with different loads can be used.

D. F. TWISS.

Pigment reinforcement of reclaimed rubber. H. A. WINKELMANN and E. G. CROAKMAN (Ind. Eng. Chem., 1928, 20, 134—137).—"Whole tyre" reclaim (containing 57% of rubber) was mixed with 5% of sulphur, and with various compounding ingredients in proportions between 2½ and 20 vols. relative to the reclaim (100 vols.). Comparison was made of the influence of "mineral rubber," whiting, barytes, *blanc fixe*, clay (including catalpo), zinc oxides (including kadox), thermatomic carbon, and carbon black on the stress-strain curve and the resistance to tearing and abrasion. Only carbon black showed any marked reinforcing action, the effect increasing with the proportion added, the resistance to tear and to abrasion being also much higher than that for any other ingredient. Measurement was also made of the plasticity of the various mixtures and of the influence of organic accelerators. The effect of an accelerator is dependent to some extent on the nature of the filler present; generally, however, with a mild ultra-accelerator improved physical characteristics are obtained in the vulcanised product.

D. F. TWISS.

Value of the rubber hydrocarbon in reclaimed rubber. W. W. VOGT (Ind. Eng. Chem., 1928, 20, 140—143).—Mixtures suitable for tyre treads, containing reclaimed rubber and of the same ultimate composition except for the proportion of new to old caoutchouc in a constant inclusive total of rubber hydrocarbon, were vulcanised to their respective technical optima in equal times. Tensile and abrasion tests indicated that the value of the caoutchouc contributed by the reclaimed material ranges from nil for small proportions up to 50% of that of new rubber for larger proportions. For the substitution of new rubber by reclaimed rubber without loss in quality it may therefore be necessary to make other adjustments in the composition of the mixture.

D. F. TWISS.

Rate of vulcanisation of reclaimed rubber. N. A. SHEPARD, H. F. PALMER, and G. W. MILLER (Ind. Eng. Chem., 1928, 20, 143—152).—When mixed with a

standard proportion of 8.3% of sulphur on the actual rubber content reclaimed rubber produced from motor covers, solid tyres, or motor tubes vulcanises much more rapidly (as judged by physical tests) than smoked sheet rubber; this characteristic causes mixtures containing new rubber together with such reclaimed rubber to vulcanise remarkably rapidly. Although residual alkali from the reclaiming process may contribute to this feature, it is not the prime cause, the identity of which is, however, still uncertain. The proportion of sulphur already in combination with the rubber hydrocarbon in the reclaimed material appears to have little influence on the rate of subsequent vulcanisation, but has a marked effect on the ageing qualities of the vulcanised product. When using rubber mixtures containing reclaimed rubber, a low concentration of sulphur and a minimum time or temperature of vulcanisation are desirable.

D. F. TWISS.

Factors in processing reclaimed rubber. P. S. SHOAF (Ind. Eng. Chem., 1928, 20, 152—155).—For the production of satisfactory reclaimed rubber, it is desirable to ensure uniform grinding of the scrap rubber, devulcanisation at a moderate temperature, efficient removal of any residual alkali, drying at as low a temperature as possible, and a minimum of milling and refining. Types of equipment are discussed and possible developments indicated.

D. F. TWISS.

Paint from rubber. GRAY.—See XIII.

PATENTS.

Vulcanisation of rubber. W. A. GIBBONS, Assr. to NAUGATUCK CHEMICAL CO. (U.S.P. 1,654,167, 27.12.27. Appl., 17.12.23).—Latex mixed with sulphur, zinc oxide, and an organic accelerator active at low temperatures is evaporated to dryness and heated at 100° for 40 min.

T. S. WHEELER.

Manufacture of an accelerator for the vulcanisation of rubber. W. J. KELLY (B.P. 283,679, 9.11.26).—Mercaptobenzthiazole may be obtained in a yield 95—97% of the theoretical by heating a mixture of aniline, sulphur, and carbon disulphide in an autoclave at 280—285° so that the pressure increases to between 600 and 700 lb./in.²

D. F. TWISS.

Production of an accelerator for the vulcanisation of rubber. L. B. SEBRELL (B.P. 283,661, 26.10.26).—Mercaptobenzthiazole, without undesirable by-products, is produced by heating a mixture of ammonia (gaseous or aqueous), aniline, carbon disulphide, and sulphur at a temperature between 100° and 250° and a pressure between 100 lb. and 1000 lb./in.²

D. F. TWISS.

Manufacture of rubber articles. DUNLOP RUBBER CO., and D. F. TWISS (B.P. 283,984, 17.9.26).—Aqueous dispersions of rubber, e.g., latex, with or without additional substances such as vulcanising agents, pigments, etc. are sprayed in a wet unheated condition on to an unheated former or rubber article, the latter of which may be a damp, unvulcanised deposit produced from latex. The articles so obtained are then dried and may be vulcanised.

D. F. TWISS.

Regeneration of rubber contained in the beads of used tyres, etc. SYND. FRANCO-NEERLANDAIS (B.P. 276,626, 16.6.27. Fr., 25.8.26. Cf. B.P. 269,127; B., 1927, 635).—The beads of discarded tyres are cut

into pieces and soaked in the oily distillate obtained by the dry distillation of waste rubber, *e.g.*, old rubber tubing. The treated beads are then placed in an acid, such as hydrochloric acid, which causes polymerisation of the absorbed oil and also renders the canvas wrapping easily removable from the hard core. The rubber present in the canvas undergoes regeneration by this treatment; the cores can be distilled for the treatment of further beads.

D. F. TWISS.

Vulcanisation of rubber. GOODYEAR TIRE & RUBBER Co., Assecs. of J. TEPPEMA (B.P. 265,919, 16.11.26. U.S., 12.2.26).—See U.S.P. 1,637,790—1; B., 1927, 789.

Manufacture of material for use in repairing punctures in the air-tubes of pneumatic tyres and other pneumatically inflatable articles. R. SURRIDGE (B.P. 284,095, 17.2.27).

XV.—LEATHER; GLUE.

PATENTS.

Cleaning of leather goods and furs. ACHILLE SERRE, LTD., and J. B. ARGENT (B.P. 283,709, 11.12.26).—Articles of leather or fur are placed in a drum with a suitable quantity of a dry, powdered, abrasive medium, *e.g.*, hardwood dust, with or without fine sand. A small proportion of a non-inflammable solvent may be added. Part of the periphery of the drum is perforated and provided with a movable cover, or covers, by means of which the perforations are closed for cleaning the goods and opened to allow the sawdust and sand to be separated from the articles.

D. WOODROFFE.

Dissolution and reprecipitation of keratins. M. BERGMANN, Assec. of H. KOESTER (G.P. 445,503, 1.2.25).—Keratin (horns, hoofs, feathers, hair, or wool), with or without a previous treatment with alkali, is stirred with an ammoniacal solution of copper or nickel oxide until dissolved. It may then be precipitated by acids or acid salts in a form suitable for production of threads or artificial masses, formaldehyde or tanning materials being added if desired. The solution may be mixed with a cuprammonium cellulose solution and the mixture spun into threads which differ considerably from other artificial silks in mechanical and dyeing properties.

C. HOLLINS.

Humic acid derivatives (G.P. 443,339).—See II. **Condensation products of arylamines** (B.P. 266,358).—See III. **Deflocculation of colloids** (U.S.P. 1,655,045).—See X.

XVI.—AGRICULTURE.

Microbiological analysis of soils. W. BENECKE and H. SÖDING (Z. Pflanz. Dung., 1927, 10A, 129—159).—The methods of Mitscherlich and of Christensen for determining the nutrient content of soils are extended. The method is based on the increased growth of algae and fungi on series of artificial media to which known amounts of soil are added. The nutrient values of various soils as indicated by this method vary somewhat with the organism used and with the duration of the experiment. Results differ from those obtained in the Mitscherlich process for soils of high organic content.

In mineral soils the two methods agree in differentiating lacking nutrients in soils, but do not indicate the same quantitative fertiliser requirement. Differences in this respect are attributed in part to results of sterilisation during the experiment and the somewhat abrupt changes in p_H value occurring during the vegetation period in some instances. In general, the relative nitrogen and phosphate contents of soils as indicated by the growth of *Aspergillus niger* under the conditions described, agree more closely with the results of chemical analyses than with values obtained by the Mitscherlich method.

A. G. POLLARD.

Soil microbiology. III. Fixative power of soil. S. WINOGRADSKY and J. ZIEMIECKA (Ann. Inst. Pasteur, 1928, 42, 36—62).—A method is described for determining the nitrogen-fixing power of organisms present in the soil which consists in determining (1) the growth obtained from 1 g. of the soil on a plate of a selective medium of silica gel and (2) the total growth obtained spontaneously in a sample of the soil.

W. O. KERMACK.

Optimum soil reaction of the sugar beet. O. ARRHENIUS (Ind. Eng. Chem., 1928, 20, 219).—The relative yield of sugar beet, as determined by pot experiments and also by field investigations over a wide area, reaches a maximum for soils which are neutral or slightly alkaline (p_H 7.0—7.5).

F. R. ENNOS.

Effect of fertiliser constituents on the eye-spot disease of sugar-cane. H. A. LEE and J. P. MARTIN (Ind. Eng. Chem., 1928, 20, 220—224).—During the winter months when conditions are favourable for infection of the sugar cane with eye-spot disease, applications of nitrogenous fertilisers tend to increase considerably the degree of infection, and should be avoided at this time in fields subject to the disease. The use of phosphoric acid or of materials containing potash does not result in increased eye-spot, and in the case of the latter appears to diminish it slightly.

F. R. ENNOS.

Relation between water and potash in plant production. F. W. MORSE (J. Agric. Res., 1927, 35, 939—946).—Pot experiments with millet and soya bean were made using soils starved of potash. Water supply was maintained at three rates, low, medium, and high. An additional series of pots received potassium sulphate. In this second series millet was nearly indifferent to the varying water supply, whilst in the first series water was a limiting factor. Soya beans were about equally affected by both potash and water.

H. J. C. HINES.

Growth of *Bacillus radicicola* on artificial media containing various plant extracts. F. E. ALLISON (J. Agric. Res., 1927, 35, 915—923).—The addition of water extracts of various plants caused marked increase in the growth of a red clover strain of the nodule organism on a sugar-salt medium. Both leguminous and non-leguminous plant extracts were used with success, wide variation occurring between different parts of the same plant. Extracts of legume roots were the most stimulating.

H. J. G. HINES.

PATENTS.

Manufacture of a dry and non-hygroscopic fertiliser from the vinasses of distilleries and sugar factories.—SELBI (Soc. d'Exploit. de Licences de

BREVETS IND.) (B.P. 279,022, 22.4.27. Fr., 16.10.26).—The preheated vinasses mixed with a distributing carrier, *e.g.*, sawdust, is stirred up with an equal weight of preheated superphosphate in a mixer at 60–70° until homogeneous. The product is then dried at 125–130°, crushed, and screened. F. R. ENNOS.

Bactericides and fungicides for treatment of seeds. CHEM. FABR. L. MEYER (G.P. 443,507, 13.3.23).—"Chloromethyl alcohol" or "fluoromethyl alcohol" the mixture of products obtained by interaction of 4 pts. of 40% aqueous formaldehyde and 6 pts. of 40% hydrochloric or hydrofluoric acid, is used for treating seeds. These are pickled for 15 min. in a 0.25–0.33% solution of the halogen compound alone or mixed with 5% of mercuric chloride, and are then covered for 8 hrs. C. HOLLINS.

Manufacture of agents for protection of plants and destruction of parasites. CHEM. FABR. DR. HEPPES & Co., G.M.B.H., Assees. of J. B. CARPZOW (G.P. 442,738, 14.9.24).—Salt- or fresh-water slimes, consisting of colloidal silicic acid and vegetable cells of micro-organisms, are dried at low temperature, mixed with insecticides *etc.*, dried, and powdered. The colloid adsorbs copper salts, arsenic compounds, formaldehyde generators (*e.g.*, hexamethylenetetramine), chlorine generators (*e.g.*, bleaching powder, trichloroethylene), or oxygen generators (*e.g.*, perborates), and in the last three cases liberates formaldehyde, chlorine, and oxygen, respectively, by catalytic action. Unlike humus extracts, the slime material does not clog the leaf-pores. C. HOLLINS.

Parasiticide and disinfectant. E. MERCK (Swiss P. 118,144, 3.6.25. Ger., 21.7.24. Addn. to Swiss P. 108,018. Cf. G.P. 401,413; B., 1925, 187).—Copper salts of cyclic organic compounds, *e.g.*, copper naphthenate, salicylate, or resinate, are used instead of copper salts of fatty acids. L. A. COLES.

Means for combating animal pests. I. G. FARBENIND. A.-G. (G.P. 443,391, 10.1.22).—The mixtures of pyridine bases obtained from acetaldehyde and ammonia or amines are efficient substitutes for nicotine. The mixture consisting essentially of 2-methyl-5-ethylpyridine is very effective against worms, caterpillars, *etc.* The pyridine base or its tannate or resinate is dissolved or suspended in water, to which magnesia, alumina, calcium carbonate, copper hydroxide, and soaps, resin salts, ligninsulphonic acid, *etc.*, may be added. The *N*-methyl and *N*-ethyl derivatives are for some purposes still more effective. C. HOLLINS.

Means for destroying animal pests. K. RÜLKE (G.P. 443,612, 25.6.22).—Introduction of an amino- or substituted amino-group into the pyridine or quinoline molecule increases the insecticidal powers of these bases. 2-Amino- or 2-ethylamino-pyridine and 2-aminoquinoline are recommended against plant-lice. Amino-derivatives of heterocyclic bases generally are claimed, alone or with soaps, saponin, alcohols, ketones, *etc.* and in solution. C. HOLLINS.

Products containing arsenic for combating plant pests. I. G. FARBENIND. A.-G. (Swiss P. 120,424–6, 8.4.25. Ger., 10.4.24. Addn. to Swiss P. 116,298).—The product contains aluminium compounds in place of

the iron compounds in the product described previously, and is prepared, *e.g.*, by stirring arsenic trioxide, aluminium sulphate, and calcium carbonate in the presence of a little water, and drying the product. L. A. COLES.

Insecticides. I. G. FARBENIND. A.-G., Assees. of K. BRODERSEN and W. EXT (G.P. 442,432, 11.9.23).—Additive compounds of phenols with quinones or amines have strong insecticidal action. *E.g.*, the compound from *p*-chlorophenol and benzoquinone at 10% dilution kills in 15 min. even the resistant beet-leaf bug, *Zosmenus capitatus*, Wolff, and small ants, *etc.* More certain, but less rapid, are the compounds from quinol and *p*-toluidine, 3:5-dinitro-*o*-cresol and pyridine, chlorodinitrophenol and pyridine, or trichlorophenol and aniline. C. HOLLINS.

XVII.—SUGARS; STARCHES; GUMS.

PATENTS.

Halogen-calcium-starch preparations. HENKEL & CIE., G.M.B.H. (B.P. 276,340, 16.8.27. Ger., 17.8.26).—The halogen-calcium-starch preparations described in B.P. 244,708 (B., 1926, 561) are treated with 50–80% alcohol to remove the excess of calcium halide. After removal of the solvent by filtration the product is dried at 45°, in which form it is capable of yielding a size with cold water which retains its covering and spreading power after being left unused for several days. F. R. ENNOS.

Fertiliser from vinasses (B.P. 279,022).—See XVI.

XVIII.—FERMENTATION INDUSTRIES.

Abnormal wines. FONZES-DIACON (Ann. Falsif., 1928, 21, 17–19).—The low temperatures and excessive rainfall in parts of France during 1927 retarded the maturing of the grapes and resulted in wines of lower alcoholic strength than normal. With the usual criteria some of these wines might be considered to be watered, but the "tartaric acid index" (B., 1928, 31) shows them to be merely abnormal. J. R. NICHOLLS.

Wines from grapes attacked by *Eudemis*. HUGUES (Ann. Falsif., 1928, 21, 19–20).—The conclusions drawn by Fabre and Bremond on this subject (B., 1928, 104) are criticised. J. R. NICHOLLS.

Methyl alcohol in spirits and tinctures. R. VIVARO (Ann. Falsif., 1928, 21, 22–24).—The methyl alcohol is oxidised to formaldehyde, which is condensed with hydroxylamine. The formaldoxime is decomposed and the resulting hydrocyanic acid is detected. The largest and most constant proportion of formaldehyde is obtained by boiling with potassium dichromate and sulphuric acid, when under the conditions specified, 10–15% of the theoretical quantity is obtained. 50 c.c. of the alcoholic solution is distilled, 20 c.c. of distillate being collected and mixed with 20 g. of powdered potassium dichromate and 200 c.c. of 10% sulphuric acid. When the dichromate has dissolved the mixture is distilled, the first 40 c.c. of distillate (containing only acetaldehyde) being discarded. The next 120 c.c. are mixed with 1 g. of hydroxylamine hydrochloride, neutralised to phenolphthalein, and 5 g. of sodium carbonate are added. After boiling for $\frac{1}{2}$ hr. with a reflux condenser the

solution is acidified with sulphuric acid and distilled into sodium hydroxide solution. The hydrocyanic acid is characterised by the ferrocyanide reaction. Furfuraldehyde, if present, must be removed by means of *m*-phenylenediamine hydrochloride or aniline phosphate.

J. R. NICHOLLS.

Relation of temperature to rate and type of fermentation, and to quality of commercial sauerkraut. H. B. PARMELE, E. B. FRED, W. H. PETERSON, J. E. MCCONKIE, and W. E. VAUGHN (*J. Agric. Res.*, 1927, **35**, 1021—1038).—Definite heat production during the fermentation of sauerkraut is indicated. The temperature of the fermenting mass governs the rate of acid production. When the temperature of the shredded cabbage is 65—75° acid production is rapid in the early stages, and becomes slow later. Below 65° the fermentation and acid production have a more uniform rate. Best-quality sauerkraut is produced under the latter conditions. The degree of fermentation is most suitably measured by means of titratable acid determinations. Measurements of p_H are less satisfactory for the purpose, being largely influenced in the initial stages of fermentation by the carbon dioxide produced. The process of fermentation is accomplished by a sequence of micro-organisms, including at least two distinct types of lactic acid-producing bacteria. Bacterial numbers may be roughly determined during the fermentation by observations of the rate of reduction of methylene-blue.

A. G. POLLARD.

PATENTS.

Production of absolute alcohol. E. C. R. MARKS. From U.S. INDUSTRIAL ALCOHOL Co. (B.P. 283,701, 29.11.26).—A continuous process for obtaining absolute alcohol from dilute alcohol (beer) is described. The beer is first distilled to give 95—96% alcohol vapours in the upper part of the rectifying unit, fusel oil being run off into a separate rectifying column from which water and fusel oil are separately drawn off, whilst alcohol vapours pass on to a dehydrating column. Here the 95—96% alcohol from the beer still is dehydrated by means of benzene (etc.), the benzene layer of the condensate being returned to the dehydrating column; the aqueous layer is diluted with water to recover more benzene and the wash waters (25% alcohol) are returned if desired to the beer still. C. HOLLINS.

Ointments (G.P. 443,756).—See XX.

XIX.—FOODS.

Different chemical constituents of the crust and crumb of Roman wheat bread. L. SETTIMI (*Annali Chim. Appl.*, 1928, **18**, 19—31).—Analyses show that the carbohydrates and nitrogenous substances in the crust and crumb of wheaten bread exhibit markedly different molecular constitutions. The crust contains greater proportions of degraded starch compounds and degraded protein derivatives than the crumb, and hence should have the greater nutritive value.

T. H. POPE.

Cheshire butters and cheeses of low Reichert-Meissl value. H. LOWE (*Analyst*, 1928, **53**, 89—90).—Samples of the butter and cheese were examined during November, and found to have low Reichert-Meissl

values, viz., 22.9 and 23.4 for two cheeses, and 22.6, 23.9, and 23.5 for three butters. A gallon sample of milk was churned in the laboratory, and the resulting butter fat had a Reichert-Meissl value of 22.1. Similar results had been obtained in previous years for November and December samples.

D. G. HEWER.

Accurate determination of the protein content of butter. B. J. HOLWERDA (*Chem. Weekblad*, 1928, **25**, 102—103).—A micro-method and apparatus are described, in which 1 g. of butter is treated with 1 c.c. of 50 vol. % sulphuric acid, the mixture kept for at least 30 min. at 40°, and then extracted twice with light petroleum to remove the fat. The residue is then treated in a micro-Kjeldahl apparatus and the nitrogen content determined in the usual way. The results are more consistent than those obtained by the macro-method, and experience shows that butter is usually sufficiently homogeneous for a 1 g. sample to be representative.

S. I. LEVY.

Determination of salt in butter and margarine. G. VAN B. GILMOUR (*Analyst*, 1928, **53**, 34).—A mixture of 10 g. of the melted butter with 20 c.c. of industrial methylated spirits is heated to the b.p., 40 c.c. of water are added, and the whole is mixed without reheating, filtered, and 30 c.c. of the filtrate are titrated with 0.1N-silver nitrate solution; the number of c.c. multiplied by 0.119 (assuming a water content of 15%) gives the percentage of salt. The method is satisfactory even in the presence of emulsifying agents.

D. G. HEWER.

Arsenic in New Zealand-grown apples. R. L. ANDREW (*New Zealand J. Sci. Tech.*, 1927, **9**, 206—209).—Apples sprayed with lead arsenate according to the usual New Zealand practice were picked at varying intervals from the last spraying and examined for arsenic. The peeled fruit (including core and seeds) was treated separately and the quantity of arsenic did not exceed 1/700 grain per lb. of whole fruit, and in most cases was negligible. The peel (including stem and calyx) contained more, but with an interval of 12—15 days between the last spraying and picking the total arsenic in the apple did not exceed 1/100 grain per lb. Ordinary wiping with a dry cloth reduced the arsenic but little, except where patches of dry spray were visible. The arsenic appeared evenly distributed over the whole skin, there being no accumulation at the stem or calyx.

J. R. NICHOLLS.

Relation of atmospheric humidity to deterioration of evaporated apples in storage. C. W. CULPEPPER and J. S. CALDWELL (*J. Agric. Res.*, 1927, **35**, 889—906).—Dried samples of five varieties of apples were stored at 20—30° under conditions such that atmospheric humidity ranged from 0 to 100%. At relative humidities above 80.5%, fungal growths appeared rapidly; between 47% and 80.5% there was no fungal growth, but changes occurred which made the fruit unmarketable before the end of the storage period. At 18.5% R.H. or less, evaporated apples maintained their original qualities indefinitely. Pretreatment of the samples with sulphur dioxide or sodium chloride, or by heating in steam at 80°, delayed deterioration at the higher

humidities, sulphur dioxide being the most effective reagent.

H. J. G. HINES.

Occurrence of acetaldehyde in Bartlett pears and its relation to pear scald and breakdown. C. P. HARLEY and D. F. FISHER (J. Agric. Res., 1927, **35**, 983—993).—A definite relationship exists between the amount of acetaldehyde in pear tissue and the severity of attacks of both scald and core breakdown, all pears having more than 14 mg. of acetaldehyde per 100 g. of fresh tissue being thus affected. Highest concentrations of acetaldehyde were found in the tissues within the breakdown area, and also in the flesh immediately beneath the scald. Acetaldehyde was demonstrated to be a normal metabolic product of healthy pears, and it is suggested that when local accumulations exceed limiting values, browning of the tissues occurs. The toxicity of acetaldehyde to pear tissue was shown artificially.

A. G. POLLARD.

Vitamins in canned foods. VI. Strawberries. E. F. KOHMAN, W. H. EDDY, and N. HALLIDAY (Ind. Eng. Chem., 1928, **20**, 202—204; cf. B., 1926, 213).—By means of feeding experiments on animals it is shown that strawberries are a rich source of vitamin-C, similar in this respect to tomatoes. The canning of strawberries results in no apparent loss of vitamin-C, probably on account of the effective removal of oxygen by the exhaust. The content of strawberries in vitamin-A is only one fortieth and in vitamin-B one fourth of that of tomatoes.

F. R. ENNOS.

Composition of fruit. L. H. LAMPITT and E. B. HUGHES (Analyst, 1928, **53**, 32—34).—A summary of analyses of various fresh fruits for the years 1925—1927.

D. G. HEWER.

Determination of mustard oil. P. SACCARDI and M. PIERI (Arch. Farm. sperim. Sci. aff., 1927, **44**, 1—10).—The Kuntze-Dieterich method of determining mustard oil gives results varying with the time during which the precipitated silver sulphide is dried. Experiment shows that the sulphide may undergo partial conversion into oxide or into sulphate, which may then lose sulphur dioxide and oxygen to form the oxide. Either of the two following methods gives satisfactory results: (1) 5 g. of the pounded mustard are macerated with 100 c.c. of water at the ordinary temperature for 30 min., three fourths of the liquid being then distilled off and made up to 100 c.c. Of this solution, 50 c.c. are mixed with 20 c.c. of 0.5*N*-alcoholic potassium hydroxide solution and heated in a reflux apparatus on a steam-bath for 30 min. The excess of alkali is determined by titration. For ordinary commercial mustard containing 0.71—0.87% of oil, the extract from 5 g. of the mustard requires 0.0224—0.027 g. of potassium hydroxide, or 0.78—0.96 c.c. of the 0.5*N*-solution for saponification. (2) 10 g. of the mustard flour are macerated for 12 hrs. with water, the mass being separated on a dry filter and washed into a 100 c.c. flask; the liquid is defecated with neutral lead acetate, de-leaded with ammonium carbonate, made up to volume, and filtered. The dextrose in 10 c.c. of the filtrate is determined by means of Fehling's solution, the percentage of dextrose, calculated on the mustard, having the mean value

0.95; for the non-defecated extract 0.94% was obtained.

T. H. POPE.

Biological values of certain types of sea food. II. Vitamins in oysters (*Ostrea virginica*). D. B. JONES, J. C. MURPHY, and E. M. NELSON (Ind. Eng. Chem., 1928, **20**, 205—210).—To convert oysters into a homogeneous, palatable form which would not deteriorate during the course of the experiments, they were drained, frozen, and the frozen product finely ground. Dehydration at 40° under reduced pressure could not be employed as it resulted in the loss of nearly all the vitamin-B properties and a considerable proportion of those of vitamin-A. The content of the frozen oysters in vitamins-A, -B, and -D, as determined by feeding tests on albino rats, compared favourably on the dry basis with that of other vitamin-containing foods. Oysters are deficient in vitamin-E. Some evidence was obtained suggesting that there may be a seasonal variation in the vitamin content of oysters.

F. R. ENNOS.

Fermentation of sauerkraut. PARMELE and others. —See XVIII.

PATENTS.

Manufacture of bread. H. A. KOHMAN (U.S.P. 1,655,707, 10.1.28. Appl., 12.12.24).—The flour, yeast, and other ingredients of the batch, including an oxyhalogen acid compound and a persulphate, e.g., 0.0015% of potassium iodate and 0.075% of potassium persulphate based on the weight of flour used, are vigorously mixed by mechanical means. Without setting the dough aside for any fermentation period, it is at once subdivided, proofed, and baked in the usual way.

F. R. ENNOS.

Manufacture of leavened bread. A. K. EPSTEIN (U.S.P. 1,657,379, 24.1.28. Appl., 14.12.25).—The dough batch is mixed with sufficient yeast-assimilable carbohydrate to enable the yeast to carry on fermentation, with a water-soluble, yeast-assimilable substance containing organic nitrogen, e.g., carbamide, and with potassium chlorate and magnesium sulphate. The amount of yeast used may be either the same as that normally required, in which case the fermentation will be more readily completed, or less than that needed to ferment the dough completely in the ordinary mixture, the fermentation in this case being allowed to continue for the usual period.

F. R. ENNOS.

Bread improver. A. H. FISCHE, Assr. to RUMFORD CHEMICAL WORKS (U.S.P. 1,657,116, 24.1.28. Appl., 7.3.27).—A mixture of powdered gum tragacanth and iodic acid, with or without flour, is added to the dough batch.

F. R. ENNOS.

Removal of the cerealin from grain. S. STEINMETZ (B.P. 259,989, 15.10.26. Ger., 19.10.25).—The grain after soaking is strained from the excess of water and subjected to pressure by beater blades in a non-ventilated drum, whereby heat is produced by friction of the grains, which serves to soften both the cellulose and the cerealin layers. The husks are then removed and part of the cerealin layer is washed off by water under pressure. The grain finally passes to a polishing drum, in which it is again subjected to pressure and heating by friction to free it completely from cerealin and to dry it.

F. R. ENNOS.

Preservation of meat. W. H. HOBBS and A. LANE (B.P. 283,626, 11.10.26).—The meat is placed in a closed chamber maintained at 0–1°, whilst a current of air, cooled to the same temperature and previously sterilised by passage through a solution of thymol in acetic acid, is introduced in the form of jets from a travelling pipe at the bottom of the chamber so that it circulates around and between and impinges against the meat, and is finally withdrawn at the top. The moisture content of the air in the chamber should be 62–66%, and, if necessary, the humidity of the circulating air may be reduced by passing over a suitable hygroscopic material, e.g., calcium chloride, and then through a layer of granular thymol prior to introduction into the meat chamber.

F. R. ENNOS.

Preparation of a soluble gum composition. A. LEO (B.P. 283,657, 18.10.26).—To standard pectin is added a mixture of finely-powdered citric acid and sodium bicarbonate containing an excess of the former. On addition of this composition to the boiling fruit juice to be jellified, the carbon dioxide evolved disintegrates the pectin and causes it to pass into solution.

F. R. ENNOS.

Conservation of animal foodstuffs. W. K. GÜNTHER (B.P. 284,130, 7.6.27).—The foodstuff, e.g., fish, is enclosed in a thin textile web and packed in sphagnum (white moss), which has been partially saturated with water cooled by ice and salt.

F. R. ENNOS.

Wrappers for foodstuffs (B.P. 283,751).—See V.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Casein glycerophosphate. J. J. L. ZWIKKER (Pharm. Weekblad, 1928, 65, 134–136).—Analysis of Sanatogen shows it to consist approximately of 5% of sodium glycerophosphate and 95% of sodium caseinate; the latter is somewhat more alkaline in reaction than the sodium caseinate prepared according to the British Pharmaceutical Codex.

S. I. LEVY.

Preparation of guaiacolsulphonic acid from *p*-dichlorobenzene. J. S. TURSKI, A. PIOTROVSKI, and S. VINAVER (Przemysł Chem., 1927, 11, 365–370; Chem. Zentr., 1927, ii, 809).—*p*-Dichlorobenzene (1 g.-mol.) is nitrated with a mixture of 90% of nitric and 10% of fuming sulphuric acid at 60° to 1 : 4-dichloro-2-nitrobenzene, m.p. 53°, which with methyl-alcoholic potash at 140° and 7 atm. in 5 hrs. yields *p*-chloro-*o*-nitroanisole, m.p. 97°. This, distilled with superheated steam, is reduced with iron filings and hydrochloric acid to *m*-chloro-*o*-anisidine, m.p. 80°, and the diazo compound decomposed in presence of copper sulphate, yielding chloroguaiacol, b.p. 236–241°, m.p. 37°. This, when heated with sodium sulphite at 175–180° for 15–20 hrs., yields 92 g. of guaiacolsulphonic acid (1-hydroxy-6-methoxybenzene-3-sulphonic acid), obtained by evaporation of the acidified product and extraction with alcohol.

A. A. ELDRIDGE.

Determination of iodine in thyroid gland preparations. E. SCHULEK and A. STASIAK (Pharm. Zentr., 1928, 69, 113–115).—The material is heated with potassium hydroxide in a nickel crucible, the melt dissolved,

the filtered solution oxidised with hypochlorite, and iodate determined by addition of potassium iodide and titration with thiosulphate. Only 0.1 g. of the material is taken, and the heating with potassium hydroxide (2.5 g.) requires only 10 min.

S. I. LEVY.

Analysis and chemistry of neoarsphenamine. A. E. JURIST and W. G. CHRISTIANSEN (J. Amer. Chem. Soc., 1928, 50, 191–196; cf. B., 1926, 27, 767).—Both free and combined formaldehyde-sulphoxylate reduce iodine in neutral solution. Consequently, when neoarsphenamine is oxidised with excess of iodine, the residual iodine reduced with sodium arsenite, the solution acidified, and the sulphate precipitated as barium sulphate, the sulphur thus determined is usually greater than that given by the Elvove method. If x be the free sulphoxylate sulphur, y the combined sulphoxylate sulphur, A the sulphur found by the above arsenite method less free sulphate sulphur, and B the iodine titration in neutral solution less the arseno-group iodine equivalent, x and y can be evaluated from the equations $x + y = A$; and $x/0.8 + y/1.6 = B$. By analysis of mixtures, synthetically prepared, containing sulphonic acids, it is shown that the difference between total sulphur and sulphur oxidisable by iodine in alkaline solution is due to nuclear sulphur introduced in the preparation of the arsphenamine base from which the neoarsphenamine is obtained, and not to sulpharsphenamine. Comparative analyses of a number of neoarsphenamines indicate the probable existence of two types of combination between arsphenamine base and sodium formaldehyde-sulphoxylate, one being of the *N*-methylene type, the other resembling double-salt formation. In addition, there is present in neoarsphenamine sulphur other than sulphoxylate, derived from some subsequent chemical change, possibly oxidation.

F. G. WILLSON.

Determination of arsenic in arsenobenzenes. A. KIRCHER and F. VON RUPPERT (Pharm. Zentr., 1928, 69, 17–18).—The authors have carried out comparative tests with their own method (A., 1924, ii, 59; B., 80), that of Stollé and Fechtig (Z. angew. Chem., 1927, 40, 1154), and that of De Myttenaere (B., 1924, 32), and find that the differences are negligible. The authors' method gave results slightly above those obtained by the other methods.

S. I. LEVY.

Determination of chloral in chloral syrup. M. FRANÇOIS (J. Pharm. Chim., 1928, [viii], 7, 54–57).—Determination of chloral in chloral syrups by the author's method (B., 1918, 166 A) gives satisfactory results. The criticisms of Andron (Bull. Soc. Pharm. Bordeaux, 1926, 64, 199) are shown to be invalid. E. H. SHARLES.

Determination of citral and similar aldehydes [in essential oils]. L. G. RADCLIFFE and W. J. N. SWANN (Perf. Ess. Oil Rec., 1928, 19, 47–51).—The reaction of thiosemicarbazide with aldehydes and ketones (Neuberg and Neiman, A., 1902, i, 572) has been studied quantitatively, particularly with reference to the determination of citral in lemon oils. The following method gives values for citral slightly higher than those obtained by the hydroxylamine method of Bennett. 2–3 g. of the oil are heated on a water-bath with 0.8 g.

of thiosemicarbazide and 50 c.c. of alcohol until most of the thiosemicarbazide is dissolved. The mixture is evaporated to dryness, boiled with 150 c.c. of carbon disulphide, and filtered. The solid, unchanged thiosemicarbazide is washed, dried, and weighed, and the amount of citral thiosemicarbazone formed can be calculated. By the use of suitable solvents the method is applicable to other aldehydes and ketones, i.e., the solvent must remove all substances except the excess of thiosemicarbazide. The method may also be employed for the detection of traces of impurities in commercial aldehydes and ketones. After separating the excess of thiosemicarbazide the presence of any impurity is indicated by the depression of the m.p. of the thiosemicarbazone remaining after removal of the solvent. In a sample of commercial citral the m.p. of the thiosemicarbazone was depressed 10°, and it is suggested that a correlation between m.p. depression and percentage of impurities might be established. The formation and properties of metallic salts of thiosemicarbazones are discussed.

E. H. SHARPLES.

Methyl alcohol in tinctures. VIVARO. See XVIII.

PATENTS.

Preparation of pyridine derivatives. DEUTS. GOLD- U. SILBER-SCHNEIDENANSTALT VORM. ROESSLER (B.P. 259,960, 11.10.26. Austr., 17.10.25).—Chlorination of β -iodopyridines yields β -pyridyl iodochlorides, $\text{NC}_5\text{H}_4\cdot\text{ICl}_2$, from which by hydrolysis with alkali β -iodopyridines are obtainable. *3-Pyridyl iodochloride*, m.p. 128—130° (decomp.), *2-chloro-5-pyridyl iodochloride*, m.p. 104—106° (decomp.), *2-chloro-5-iodopyridine*, decomp. 200—205°, and *2-chloro-5-iodoxypyridine*, detonating at 210—215°, are described. The iodoxy-compound is prepared by distilling in steam the iodoso-derivative.

C. HOLLINS.

Production of isonaphthyridine and its derivatives. DEUTS. GOLD- U. SILBER-SCHNEIDENANSTALT VORM. ROESSLER (B.P. 259,973, 13.10.26. Aust., 17.10.25).— β -Aminopyridines are subjected to the Skraup or Doebner reaction. *2-Chloro-5-aminopyridine*, with glycerol, arsenic pentoxide, and sulphuric acid, gives *2-hydroxy-1:5-naphthyridine* [*2-chloroisonaphthyridine*], not melted at 250°; *1:5-[or iso]naphthyridine*, m.p. 72°, b.p. 112°/12 mm., is similarly obtained from *3-aminopyridine*. *2-Chloro-3-aminopyridine* condenses with pyruvic acid and benzaldehyde to form *2-chloro-6-phenyl-1:5-naphthyridine-8-carboxylic acid*, decomp. 186°. In each case cyclisation occurs *ortho* to the pyridine nitrogen.

C. HOLLINS.

Preparation of quinoline derivatives. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 282,143, 11.9.26).—4-Aminoquinolines, obtained by replacement of a halogen, alkoxy-, hydrazino-, carbonamido-, or carbazido-group in 4-position by an amino-group, are condensed with aromatic aldehydes; or the introduction of the amino-group may follow the condensation. The products are strong bactericides. The following compounds are described: *4-Chloro-2-styrylquinoline*, m.p. 117°, *4-amino-2-styrylquinoline*, m.p. 173—175°; *4-chloro-6-ethoxy-2-styrylquinoline*, m.p. 120°, and the *4-amino-compound*, m.p. 210° (*glycollate*, m.p. 237°); *4:6-di-*

ethoxy-2-styrylquinoline (from the 4-chloro-6-ethoxy-compound and sodium ethoxide); *4-phenylhydrazino-2-styrylquinoline* (from 4-chloro-2-styrylquinoline and phenylhydrazine), *4-phenylhydrazino-6-ethoxy-2-styrylquinoline*: *4-chloro-2-methyl- β -naphthaquinoline*, m.p. 200°; *4-chloro-2-styryl- β -naphthaquinoline*, m.p. 128°, *4-amino-2-styryl- β -naphthaquinoline*, m.p. 226—227°; *4-amino-6-ethoxyquinoline*, m.p. 195°; *4-amino-6-ethoxy-2-m-nitrostyrylquinoline*, m-aminostyryl compound, m.p. 182°; *2-styrylquinoline-4-carboxylic acid* (methyl ester, m.p. 98°; ethyl ester, m.p. 77°; amide, m.p. 274°; hydrazide, m.p. 215°; azide; urethane, m.p. 202°); *4-diethylamino-6-ethoxy-2-styrylquinoline*, m.p. 178° [*glycollate*, m.p. 214—216° (decomp.)], *4-diethylamino-compound*, m.p. 103°; *4-diethylamino-6-ethoxyquinoline*, m.p. 74°.

C. HOLLINS.

Manufacture of 2-phenylquinoline-4-carboxylic acid [atophan]. A. J. STEPHENS. From R. von WULFING (B.P. 283,822, 23.9.27).—By the use of the compound of isatin with sodium or potassium hydrogen sulphite (sodium compound contains 52.9% of isatin) for condensation with acetophenone a very pure atophan, m.p. 212—213°, is obtained.

C. HOLLINS.

Manufacture of crystallised arsenobenzenes. A. ALBERT (B.P. 259,245, 4.10.26. Ger., 3.10.25).—Pure crystalline arseno-compounds are obtained by reducing with hypophosphorous acid (or salts) an arsenic acid, or arsenoxide, or dihalogenoarsine, containing the grouping, $\text{As}\cdot\text{Ar}\cdot\text{COR}$, where R is hydrogen or alkyl, or the hydrazones, semicarbazones, etc. of these. *3-Hydroxybenzaldehyde-4-arsinic acid*, prepared from 3-nitro-4-aminobenzaldehyde by Bart's reaction followed by reduction of the nitro-group and diazotisation, is reduced in the form of its semicarbazone, or of the *dichloroarsine*, to *2:2'-dihydroxy-4:4'-dialdehydoarsenobenzene*. *3-Hydroxyacetophenone-4-arsinic acid* semicarbazone, or the *dichloroarsine*, or the *arsenoxide*, gives *2:2'-dihydroxy-4:4'-diacetylarsenobenzene*, decomp. above 200°.

C. HOLLINS.

Preparation of organo-mercury compounds. F. HEIN and K. WAGLER (G.P. 441,666, 13.8.24).—Organo-mercury salts are treated with metals (copper, silver, iron, nickel, cobalt, zinc) or their alloys in presence of basic nitrogen compounds (ammonia, amines, pyridine bases and homologues) with or without other solvents. Benzyl mercurichloride in pyridine is treated for 1—2 hrs. with copper turnings or warmed for 1 hr. with copper gauze; the pyridine is distilled off in a vacuum and copper chloride removed by means of aqueous ammonia, leaving mercury dibenzyl, $\text{Hg}(\text{CH}_2\text{Ph})_2$, in 90% yield; mercury is recovered by heating the copper in hydrogen. Mercury diphenyl, m.p. 125°, is obtained by treating phenyl mercurichloride with zinc turnings in alcoholic pyridine, or diethylamine or liquid ammonia. *Mercury 2:2'-dinitrodiphenyl*, m.p. 204.5—205°, and *mercury tetramethyldiaminodiphenyl*, m.p. 167—168°, are similarly prepared. The products find pharmaceutical application.

C. HOLLINS.

Benzylresorcinols. A. R. L. DOHME, Assr. to SHARP & DOHME (U.S.P. 1,658,229, 7.2.28. Appl., 9.10.23).—See B.P. 223,190; B., 1925, 378.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Experiments with hyposulphite and Rongalite. F. BÜRKI (Phot. Korr., 1928, 64, 16—17).—A 0.1% solution of hyposulphite acts as a developer, giving a slight fog. A 1% solution acts more quickly but with pronounced fog, whilst more concentrated solutions give mostly fog. If Rongalite, the addition product of hyposulphite and formaldehyde, is used the solution does not act as a developer, and the latent image, after treatment with this solution, can still be developed with a normal developer. Pre-treatment of the latent image with formaldehyde accelerates the subsequent developing action of hyposulphite or of an ordinary developer. Selenium is soluble in a solution of hyposulphite or of Rongalite, giving a neutral, colourless solution, which can be used for toning papers.

W. CLARK.

Constitution of the flavinduline desensitisers. G. KÖGEL (Phot. Korr., 1927, 63, 193—195).—The desensitising group is considered to be the azomethine group $>C:N\cdot$, of which there are two in flavinduline.

W. CLARK.

Development of Daguerre plates by cathode pulverisation. G. SIMON (Compt. rend., 1928, 186, 139).—Daguerre plates (polished silver lightly treated with iodine) may be developed by exposure for a fraction of a second to the pulverisation obtained from a pure or an amalgamated copper cathode, in place of exposure to mercury vapour. Gold or platinum cathodes gave negative results.

J. GRANT.

Hypothesis of the latent image. A. P. H. TRIVELLI (Z. wiss. Phot., 1928, 25, 65—78).—See B., 1928, 107.

Fog correction of photographic blackening. H. A. PRITCHARD (Z. wiss. Phot., 1928, 25, 79—113).—See B., 1927, 957.

PATENTS.

Light-sensitive paper etc. A. H. MEURSING and E. J. GRATAMA, ASSEES. of T. RODY (B.P. 274,075, 1.7.27. Ger., 7.7.26).—For producing a sensitive velour-like material such as paper, linen, silk, etc. an adhesive substance, insoluble in water, is applied to the base, after which fine vegetable or animal fibre dust is applied. The fibre dust is rendered sensitive before or after application, for example by bathing the paper etc. in a silver nitrate solution followed by a solution containing ammonium bromide and potassium iodide.

W. CLARK.

Colour photography. W. T. TARBIN (B.P. 283,765, 17.3.27. Ger., 1.2.27).—In a method of colour photography using a number of superimposed layers each sensitive to a different spectral region, the layer sensitive to the blue is arranged so that in exposure it is the farthest from the source of light. The composite plate may be built up as follows. A glass plate is coated with a red-sensitive stripping layer in contact with which is a non-stripping green-sensitive layer on a thin celluloid support, whilst the blue-sensitive layer is coated on glass, the layer being in contact with the back of the celluloid support of the green-sensitive layer.

W. CLARK.

Production of a colour photograph or film. F. E. IVES, ASSR. to C. W. FENNINGER (U.S.P. 1,655,182, 3.1.28. Appl., 29.10.20).—The process of U.S.P. 1,278,668 (B., 1918, 784A) is modified in that a blue-to-green, iron-toned image is produced at the interior surface of the colloid layer, which is then treated with potassium bromide to resensitise the silver present, and the red image is produced at the outer surface.

T. S. WHEELER.

Preparation of layers for surface-printing processes. U. OSTWALD (B.P. 262,793, 8.12.26. Ger., 8.12.25).—In the preparation of a layer for surface-printing processes, glue, gelatin, or albumin is mixed with a non-precipitating, water-insoluble, organic colloid which strengthens the particles of the glue etc. and reduces its water-absorbing properties. When dry the layer is treated with an agent which tans it on exposure to light. The glue etc. is dissolved in a suitable organic solvent, preferably acetic acid, whilst the organic colloid used may be a cellulose derivative and/or a resin in a solvent such as acetic acid. Rubber or linoleum or other elastic substance is used as the support and solutions of cellulose derivatives containing plastifiers are used as intermediate layers in cases when the main layer will not adhere firmly to the support. After washing out the solvent, sensitising with dichromate, exposing, and washing out the dichromate, the unexposed parts of the layer are rendered porous by treatment with alkaline reagents such as ammonium carbonate or an alkali carbonate.

W. CLARK.

Coloured magnesium flame (G.P. 445,181).—See X.

XXII.—EXPLOSIVES; MATCHES.

PATENTS.

Manufacture of explosives. F. W. GUTHKE (B.P. 275,228, 25.7.27. Ger., 28.7.26).—Explosives are obtained by nitrating mixtures of solid and liquid polyhydric alcohols. In particular, the explosive may contain a mixture of nitrated sorbitol and nitrated ethylene glycol with or without nitrated glycerol.

S. BINNING.

Fuse compositions and fuses for shells. CURTIS'S & HARVEY, LTD., and A. J. GRIMWOOD (B.P. 283,741, 21.1.27).—A powder for use in time rings for shell fuses consists of black powder in which all or part of the charcoal is replaced by cuprene. The powder may be compressed into brass-lined channels in rings made of aluminium or its alloy, to form a shell time-fuse which may be combined with a percussion fuse.

S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Carbon and nitrogen transformation in fresh sewage solids digestion. H. ILEUKELEKIAN and W. RUDOLFS (Ind. Eng. Chem., 1928, 20, 177—179).—The changes in carbon, nitrogen, and volatile matter during the fermentation of fresh sewage solids are indicated graphically, the results obtained with and without the addition of lime to maintain a p_H value of 7.4 being compared. It is shown that the total nitrogen content is unchanged, though a decreasing amount of volatile matter causes a rise in the percentage nitrogen

In the case of lined material there is a reduction of 60% in the volatile matter and of 30% in the carbon content during a period of 8 months. The addition of lime facilitates the decomposition of certain types of organic matter, possibly fats, with consequent increase in the volume of gas produced.

C. JEPSON.

Gases from sewage sludge digestion. W. D. HATFIELD, G. E. SYMONS, and R. R. MILLS (Ind. Eng. Chem., 1928, 20, 174—176).—The sewage from Decatur (Illinois) contains half its volume of waste from a corn products factory, and has a temperature of 20—40°. Under these conditions putrefaction is rapid and the gases evolved contain up to 1% by vol. of hydrogen sulphide. Plant and laboratory experiments have shown that the rate of gas formation reaches a maximum about 30°, and that, at 30—35°, half the volume of gas evolved is obtained from sludge deposited during the previous 24 hrs.

C. JEPSON.

Activated sludges. L. CAVEL (Compt. rend., 1928, 186, 433—436).—Determinations of the carbon and nitrogen contents of activated sludges before and after aeration for five months showed that 4.32% and 67.17% were removed, respectively. Similar determinations using the dried and heated sludge have shown that the reduction of organic matter, under the influence of aeration, refers almost exclusively to the nitrogen removed, and indicates that the sludge is de-activated by an excess of nitrogen, since when this is removed by aeration its properties are restored. Unlike that of bacterial beds, the activity of sludges depends largely on the composition of the sewage treated.

J. GRANT.

Natural and artificial black sand for demanganising water. H. LÜHRIG (Gas- u. Wasserfach, 1927, 70, 1277—1281).—The black sand, containing deposited manganese oxides, found in the alluvial bed of the Oder, occurs only in places to which air has access. When water is passed through a filter bed of this sand, part of the oxygen in the former is removed, not bacteriologically but apparently by certain oxides of manganese which give it up again in the process of demanganising water. This is borne out by the observations that the demanganising efficiency of the sand increases with use, and that demanganisation is incomplete if the concentration of manganese in the water exceeds about 45 mg. of manganous oxide per litre. By supplying additional oxygen, however, *e.g.*, as permanganate, demanganisation is complete even with such concentrations. Other per-salts, also chlorine, have the same revivifying effect on the sand, although chlorine may, in certain concentrations, exert a poisoning action. The chemistry of the demanganising process is not yet clear, but the same results are obtained with both natural and artificially prepared black sands.

W. T. K. BRAUNHOLTZ.

Stable colour standards for the determination of nitrites in water. R. DANET (J. Pharm. Chim., 1928, [viii], 7, 113—114).—For the determination of nitrites in water by the Griess method, Fuchsine S is used as colour standard. In four standard tubes are placed 0.3, 0.6, 0.9, and 1.2 c.c., respectively, of a 0.01% solution of Fuchsine S and the volume in each is made up to 10 c.c. with distilled water saturated with camphor and con-

taining 1% of acetic acid. In a similar tube are placed 10 c.c. of the water and 1 c.c. each of the sulphanilic acid and naphthylamine reagents, and the colour formed after 20—30 min. is compared with those of the standards, which represent 0.5, 1.0, 1.5, and 2 mg. of sodium nitrite per litre, respectively.

E. H. SHARPLES.

Vorce's method for determination of small quantities of phenol in impure water. F. LIEBERT and W. M. DEERNS (Chem. Weekblad, 1928, 25, 103—105; cf. B., 1925, 861).—The four stages of the method, viz., purification and concentration of the water, separation of the phenol, and colorimetric determination, have been examined to estimate the errors which may arise. Since phenol itself is readily oxidised by hydrogen peroxide in bright light, the purification should be carried out in the dark. No losses of phenol occur during concentration under the conditions laid down; the method of separation is also satisfactory. Use of corks or rubber stoppers in the apparatus for the distillation gives rise to errors, the results being too high in consequence of the presence in the phenol solution of reducing substances from the stoppers or corks. The tungsten molybdate reagent employed for the colorimetric determination is not specific for phenol, so that errors may arise from this cause; generally, however, the method is sufficiently accurate for ordinary purposes.

S. I. LEVY.

Elimination of errors in the o-tolidine method [for determination of chlorine in water]. F. R. McCURMB (J. New England Water Works' Assoc., 1927, 41, 386—398).—The colour-producing substance is shown to be a hydrogen-ion indicator giving the desired yellow when the liquid under test has pH 2.0 or less. In order to ensure this condition, the maximum amount of hydrochloric acid which can be used in making up the o-tolidine solution has been determined, and a method of preparation is recommended. A comparator set is described by which it is possible to minimise the interference due to colour, turbidity, and the effects of manganese, iron, and nitrites.

C. JEPSON.

Effect of addition of salts on the germicidal efficiency of sodium hydroxide. M. LEVINE, J. H. TOULOUSE, and J. H. BUCHANAN (Ind. Eng. Chem., 1928, 20, 179—181).—The addition of sodium chloride, sodium carbonate, and trisodium phosphate to sodium hydroxide markedly decreases the time required at 50° and 60° to effect a 99.9% removal of bacteria. Trisodium phosphate is less efficient than the other two salts, the effects of which are approximately the same. Reasons to account for these results are suggested (cf. B., 1928, 70).

C. JEPSON.

Eye protection glasses. BLOCH.—See VIII.

PATENTS.

Cleansing agent for teeth. C. H. BOEHRINGER SOHN, Assees. of H. STROBEL (G.P. 442,857, 15.5.25).—Salts which generate small amounts of acid by hydrolysis, especially organic salts, and aluminium lactate in particular, are used with or without addition of chalk, magnesium carbonate, or other acid-binding agent.

C. HOLLINS.

Disinfectant (Swiss P. 118,144).—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 13, 1928.

I.—GENERAL; PLANT; MACHINERY.

Sedimentation analysis. III. S. ODÉN and N. WINCKLER (*Tekn. Tidskr.*, 1927, 57, [Kemi 6], 16—18; *Chem. Zentr.*, 1927, ii, 962—963).—The application of sedimentation analysis to the investigation of powdered substances of technical interest is indicated.

A. A. ELDRIDGE.

Condensation of liquids as mists. M. PAILLY (*Rev. gén. Colloid.*, 1928, 5, 745—748).—The formation of mists is a result of the dilution in other gases of the vapour concerned, and the principal force acting on the droplets is surface tension which varies with the composition of the vapour and gas. Tar and oil, which are of high viscosity, are readily precipitated mechanically. Sulphuric acid mists are much more difficult to precipitate, and the problem of designing sulphuric acid chambers of the greatest efficiency can be stated in terms of mist condensation, all designs employing cooling and "shock" methods for the purpose. In the condensation of sulphuric acid mists from concentration plants the Cottrell apparatus has great advantages over coke scrubbers, with or without cooling, but requires somewhat delicate operation owing to the structure of the lead apparatus and the necessity of electrical insulation.

C. IRWIN.

Detection of phosgene in chemical fire extinguishers. E. GLASER and S. FRISCH (*Z. angew. Chem.*, 1928, 41, 263—265).—Most chemical fire extinguishers contain carbon tetrachloride which on thermal decomposition in presence of oxygen produces phosgene. A number of accidents have occurred from this cause, and it has been claimed that the addition of ethylene dibromide reduces the formation of phosgene. The authors have found that by passing a mixture of dry air and carbon tetrachloride vapour through a 30 cm. tube heated at 250° and charged with anhydrous ferric chloride and pumice, phosgene up to 45% of the theoretical yield is obtained. The resultant gases were freed from chlorine and passed into aqueous aniline, and the precipitate of diphenylurea was weighed. The reaction proceeds up to equilibrium and the residual gases freed from phosgene can be further oxidised; methyl bromide, if present, increased the yield of phosgene, but ethylene dibromide reduced it. The composition of the solutions in various fire extinguishers is given, and the use of those containing carbon tetrachloride condemned as dangerous.

C. IRWIN.

Refractories in boiler furnaces. JOHNSON and KING. **Slags from boiler furnaces.** McDOWELL and LEE.—See VIII.

PATENTS.

Furnaces. STEAM PRODUCTION CORP., Assees. of F. J. CURRAN (B.P. 275,177, 11.6.27. U.S., 31.7.26).—

Part of the fuel and air is admitted below a grate through which the mixture passes and burns above it, the upper surface of the grate being provided with upstanding "radiants" or fins which provide a hot surface for promoting combustion. Another part of the fuel and air is admitted to a helical preheating space between the furnace wall and an outer wall, then enters the furnace at an angle to promote whirling some distance above the grate; these entrances also are provided with deflector plates which become hot.

B. M. VENABLES.

Furnace. E. P. STEVENS and R. J. HIMMELRIGHT, Assrs. to AMER. ARCH CO. (U.S.P. 1,656,963, 24.1.28. Appl., 16.11.22).—A section of the furnace roof, normally sealed with the side walls, is provided with means for raising it into position for forward movement along a track extending forward of the furnace. A protective shield is located beneath the section in its forward position.

H. HOLMES.

Furnace. W. M. DUNCAN (U.S.P. 1,657,113, 24.1.28. Appl., 9.1.24).—An inclined arch constituting the front wall of the combustion chamber is built of refractory blocks each comprising a head portion, a body portion provided with a heat-radiating face in the chamber, and oppositely-arranged recesses between the head and body portions. Pairs of rods extend through these recesses and through plates carried by ribs inclined parallel to the arch, and the ribs are suspended by upright rods from transverse horizontal beams in front of the arch.

H. HOLMES.

Annealing furnaces. BRIT. FURNACES, LTD., and E. W. SMITH. From SURFACE COMBUSTION CO. (B.P. 283,767, 22.3.27).—In a heating chamber a combustion chamber is formed by a partition wall along one side and the flames pass out above the partition, over, down the other side of, and underneath a muffle or annealing box to exit ports in the floor of the main chamber which lead to a recuperator. Air from the recuperator and gas from a producer enter through separate ports in the floor of the combustion chamber, and there are also ports through the partition wall so that a portion of the burnt gases from the heating chamber can be mixed with the new fuel. All three sets of ports are controlled by sliding tiles over them. The muffle is off-centre of the heating chamber so that the space round it varies roughly according to the decrease in volume of the gases as they lose their heat.

B. M. VENABLES.

Annealing furnaces. W. G. BEESTON (B.P. 283,725, 31.12.26).—The waste gases are drawn off from one end of the furnace through several flues which lead back underneath the furnace and are individually provided with dampers, so that by regulating the latter the direction of the heating gases in the furnace may be

controlled and the heat uniformly spread. Additional air enters the furnace underneath the inlet for the main combustion gases, one method of effecting this being through a hollow firebridge which has an overhanging lip to project the flames well into the furnace and improve the distribution of heat. B. M. VENABLES.

Recuperative furnace. J. L. HOTT (U.S.P. 1,658,199, 7.2.28. Appl., 14.10.25).—The hot gases from the combustion chamber are passed through pipes arranged within the recuperator chamber, and pressure devices are provided for producing a flow of the air thus heated through a conduit into the combustion chamber. Temperature-controlled means serve to diminish the amount of air supplied to the combustion chamber without varying the flow through the recuperator chamber. H. HOLMES.

Rotary kilns for burning cement, ore, and similar materials. VICKERS, LTD., and L. D. PARKER (B.P. 284,494, 23.3.27).—The kiln has its interior formed of a number of cylindrical portions inclined to the general axis of the kiln joined by cylindrical portions not inclined to, but eccentric to, the axis, so that a longitudinal section is wave-like. B. M. VENABLES.

Rotary kilns, dryers, etc. E. ALLEN & Co., LTD., and W. J. COLES (B.P. 283,669, 29.10.26).—A rotary kiln is rotated fast enough to cause the material to cascade without using lifting devices, and means are provided to retain sufficient material in the kiln to effect the cascading, one of such means being reduced diameter of the ends. B. M. VENABLES.

Apparatus for effecting heat transfer. H. F. SMITH, Assr. to GAS RESEARCH Co. (U.S.P. 1,656,836, 17.1.28. Appl., 16.3.22).—A water heater comprises a central, vertical, copper tube which is surrounded by the water under treatment, and up which hot gases are led. To facilitate transfer of heat the tube is provided with a number of copper gauzes which extend across it and are in thermal contact with the inner side. T. S. WHEELER.

Cooling towers. K. W. BRANCZIK (B.P. 284,018, 21.10.26).—Cooling towers of large diameter or width are constructed in several sections with unoccupied spaces in between so that air has free access to any part of the interior, avoiding dead zones. The spaces are preferably radial with wider outer ends. An arrangement of distributing launders and footways is described. B. M. VENABLES.

Crushing and separating apparatus. **Crushing apparatus.** P. L. CROWE (U.S.P. 1,654,638—9, 3.1.28. Appl., 20.10.25).—(A) Fan units are enclosed in a casing mounted on a base. A conduit projects from each side of the casing and a grinding cylinder is arranged concentric with each conduit. (B) Two plates each comprising an upper portion directed downwards and inwards, and a lower portion directed downwards and outwards, are pivoted near their adjacent apices. The upper portions constitute a crushing jaw upon oscillation of the lower portions by a cam between them. H. HOLMES.

Conical crushing mill. H. ACKERMANN and W. LANGENHEIM (U.S.P. 1,656,895, 24.1.28. Appl., 5.2.27.

Ger., 6.8.25).—A seating element fixed to a shaft carries a conical crushing mantle upon a filling of hard lead or like material. Steps, some of which are undercut, are provided on the seating element. H. HOLMES.

Impact or beater mills. AMME-LUTHER WERKE BRAUNSCHWEIG DER "MIAG" MÜHLENBAU U. IND. A.-G. (B.P. 283,831, 28.10.27. Ger., 31.1.27).—An impact mill of the type where the beaters protrude between spaced bars is also fitted with an easily movable anvil. B. M. VENABLES.

Grading and separation of mixed materials. F. B. PETRIE, and PETRIE & McNAUGHT, LTD. (B.P. 283,645, 14.10.26).—Material such as towns' refuse is passed over longitudinal grizzly bars where it is pushed by cross-bars and exposed for hand sorting, then through a rotary screen and magnetic separator. Provision is made for removing the non-rolling material such as garbage. B. M. VENABLES.

Centrifugal extractor. A. R. CURTIS (U.S.P. 1,659,391, 14.2.28. Appl., 23.4.23).—The rotary basket is mounted in a vessel fitted with a lid having means for locking it in closed position. Means are also provided for starting and stopping the motor and for connecting and disconnecting the drive. The starting device for the motor is operable only when the drive is disconnected, and the connexion of the drive is possible only when the lid is closed and locked. H. HOLMES.

Centrifugal apparatus. WESTERN STATES MACHINE Co., Assocs. of E. ROBERTS (B.P. 280,167, 19.7.27. U.S., 5.11.26).—A centrifugal machine suitable, e.g., for the purification of sugar, is provided with means for rotating at a high or low speed, with a brake, and with sprays for washing. The periods of low speed for charging and of stoppage for discharging are under human control, but the periods of accelerating and high speed for separating, of spray washing, of drying, and of deceleration are under automatic (though adjustable) control. B. M. VENABLES.

Filter for fluids. C. H. CUNO, Assr. to CUNO ENGINEERING CORP. (U.S.P. 1,657,346, 24.1.28. Appl., 19.1.27).—A filtering member is provided with radial filtering passages, and with delivery passages normal to the plane of the latter, and is displaceable angularly to enable cleaning members to be engaged with it. H. HOLMES.

Rotary filters or strainers. F. W. BRACKETT (B.P. 284,403, 28.10.26).—In a strainer of the rotary-cup type, for large quantities of liquid such as condensing water, comprising a cylindrical rotating strainer with one closed end and with inlet at the open end and outlet through the circumference which is only partly submerged, buckets are provided within the strainer which lift refuse from the outlet side and carry it across to the other side where it is caught by a hood and launder and conveyed away by a separate stream of water. To preserve a good seal between the rim of the open end and the fixed structure of the screen, rollers are placed on the closed end which run over fixed rails and relieve the shaft of canting strain. B. M. VENABLES.

Vacuum-filter liquid guard. G. S. BACKUS, Assr. to OLIVER CONTINUOUS FILTER Co. (U.S.P. 1,654,717, 3.1.28. Appl., 9.12.26).—An enclosed vacuum and

pressure chamber behind the filter wall is provided with suction pipes for drawing a liquid through the wall and with pipes for supplying a gas or liquid for blasting off the filter cake. A metal plate extends across the filter in front of the blasting pipes to prevent direct impact of the fluid therein on the filter wall. H. HOLMES.

Separation of liquids of different gravity. W. LINNMANN, JUN. (U.S.P. 1,655,191, 3.1.28. Appl., 22.11.26. Ger., 8.6.26).—A tank provided with an inlet encloses a container closed at the top and open to the tank below, and an outlet pipe projects from the container. The upper part of the container communicates with the tank by a passage extending downwards to a level above the inlet end of the pipe. H. HOLMES.

Apparatus for purifying or cooling gases by treatment with a liquid. A. TOMSON (B.P. 283,682, 13.11.26).—The gas passes in one direction through an inner cylindrical casing, where it is sprayed with the liquid and returns through a surrounding annular space which is provided with a helical baffle to remove the mist of liquid. B. M. VENABLES.

Treatment of gases with liquids. M. D. MANN, JUN., Assr. to S. B. HUNT (U.S.P. 1,654,181, 27.12.27. Appl., 14.5.20).—The gases are treated with immiscible liquids in a chamber arranged between inner and outer chambers, through which is passed a temperature-controlling fluid. The liquid aggregate is introduced into the treating chamber near the top, and as it descends is separated into strata, and the gases, introduced near the bottom, bubble through the successive strata.

H. HOLMES.

Colorimeter [for liquids]. C. ZEISS, Assee. of L. LORBER (B.P. 280,552, 9.11.27. Hung., 10.11.26).—Light from a common source is divided into two beams which pass through a pair of comparator units and is viewed as a disc divided into halves of colours which are to be equalised. Each comparator unit comprises a fixed outer glass reservoir for liquid, an inner glass reservoir which can be moved (vertically) parallel to the beam of light, and a fixed glass cylinder which extends to the bottom of the inner reservoir when the latter is in its uppermost position. The bottoms of the reservoirs and the ends of the rod are all truly perpendicular to the beam of light. The comparator is suitable for estimating the amount of coloured solute even when the solvent itself is coloured and/or turbid.

B. M. VENABLES.

Viscosity tester for oils. F. B. MASON (U.S.P. 1,659,534, 14.2.28. Appl., 16.1.26).—Containers are mounted in a frame, and a receptacle is secured within one end of each container. Means are associated with each receptacle for determining the time required by liquid filling the receptacle to flow therefrom.

H. HOLMES.

Flask for laboratory purposes. H. THIENE, P. H. PRAUSNITZ, and G. SCHOTT, Assrs. to JENAER GLASWERK SCHOTT & GEN. (U.S.P. 1,659,383, 14.2.28. Appl., 18.12.26. Ger., 12.12.25).—The lower end of the flask is shaped as a frustum of a cone tapering downwards at an angle of not less than 90° to the slope of the body portion. The lower end of the frustum provides a base perpendicular to the axis of the flask, and the side of the frustum is

flattened to provide a second base oblique to the axis. H. HOLMES.

Process and apparatus for heat transfer. R. W. DAVENPORT, Assr. to CHICAGO PNEUMATIC TOOL CO. (U.S.P. 1,659,681—2, 21.2.28. Appl., [A] 25.9.25, [B] 9.10.25).—See B.P. 258,847; B., 1927, 688.

Temperature-controlling devices for furnace chambers and other apparatus. N. H. FREEMAN (B.P. 284,008, 19.10.26.).

Reversing apparatus for gas, air, and waste-gas valves of gas-fired furnaces, e.g., regeneratively heated coke ovens, metallurgical furnaces, etc. KELLNER & FLOTHMAN G.M.B.H. (B.P. 275,206, 13.7.27. Ger., 31.7.26).

Purification of liquids (U.S.P. 1,654,812).—See XXIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Low-temperature assay [of coal] in the Fischer aluminium retort. HEMPEL (Brennstoff-Chem., 1928, 9, 3—6).—The tar and liquor are collected in a water-cooled receiver the lower end of which is narrow and graduated and carries a tap. The weight of tar and liquor together is noted, and the receiver is then immersed in warm water (about 70°), whereby the tar melts and separates from the water. The latter is run off, the warming and separation are repeated twice, and the remaining tar (containing water not exceeding 0.12% of the original coal) is weighed. The method gives rather lower results for the tar yield than the older method of weighing the water and not the tar. A lignite containing 47.25%, and, after partial drying, 13.52% of water gave by the old method 23.24% and 21.48% of tar, and by the new method 21—47% and 20—63% of tar, respectively.

W. T. K. BRAUNHOLTZ.

Comparison of the methods commonly used in Germany and Holland for determining the content of volatile matter of coals. D. J. W. KREULEN (Brennstoff-Chem., 1928, 9, 2—3).—The methods in use in Germany and Holland, which differ mainly in the intensity of the flame heating the crucible, give concordant results provided that air-dried coal is used in both. The use of absolutely dry coal, which is often hygroscopic, may entail errors in weighing in a damp atmosphere.

W. T. K. BRAUNHOLTZ.

Graphitic oxide and the Brodie reaction. O. W. STOREY (Amer. Electrochem. Soc., April, 1928. Advance copy. 10 pp.).—The action of the Brodie mixture (fuming nitric acid and potassium chlorate) on Acheson graphite and on samples of lampblack calcined at various temperatures has been examined. Lampblack calcined at 1100—1200° gave a slightly oxidised product after two treatments with the Brodie mixture, but in subsequent treatments the graphitic oxide was destroyed and the whole material went into solution. By calcination at higher temperatures the lampblack became more resistant, but grey or creamy white oxidation products could eventually be obtained. Acheson graphite is still more resistant and yields a pale yellow oxidation product after about 8 treatments with the oxidising mixture.

H. J. T. ELLINGHAM.

Determination of asphalt in minerals. E. CASIMIR (Petroleum, 1928, 24, 315—318).—Two methods for the determination have been critically examined; extraction with benzene or chloroform, using the Soxhlet apparatus, was found to give higher results than treatment with a mixture of hydrochloric acid and ether (Prettner method). This was due to conversion of part of the asphalt into an ether-insoluble form by the action of the acid, and also, to a less extent, to loss of sulphur. In both methods the extracted asphalt contains ash which is carried in colloidal solution; chlorine also is present in the asphalt after acid extraction. R. H. GRIFFITH.

Determination of the tar value [of mineral oils]. H. VON DER HEYDEN and K. TYPKE (Chem.-Ztg., 1928, 52, 150).—The method suggested by Marcusson and Bauerschäfer (B., 1926, 427) gives results which, in the majority of cases, differ considerably from those obtained by the specification method, and cannot be regarded as a substitute for the latter. W. J. POWELL.

Constituents of low-temperature tar. E. BÖRNSTEIN (Nature, 1928, 121, 356—357).—A statement of priority involving a correction. A. A. ELDRIDGE.

[Constituents of low-temperature tar.] G. T. MORGAN and D. D. PRATT (Nature, 1928, 121, 357).—An acknowledgment of Börnstein's explanation (cf. previous abstract). A. A. ELDRIDGE.

Separation of the components of petroleum. VI. **Action of glacial acetic acid.** P. F. GORDON and J. MERRY (J.S.C.I., 1927, 46, 429—432 T).—After preliminary tests, crude Persian petroleum (d_{40}^{20} 0.839) was extracted with glacial acetic acid, leaving a residual oil which was filtered to remove asphaltic matter. The acid-soluble portion was fractionally precipitated by the successive addition of small quantities of water. The fractions thus obtained showed a steady increase in sp. gr. of the precipitated oil as water was added of from 0.860—0.962. The acid-insoluble oil was dissolved in ether and fractionally precipitated by successive additions of small quantities of absolute alcohol. The heaviest fractions precipitated first had approximately the same sp. gr., diminishing on further additions of alcohol. After addition of excess of alcohol, a further fraction was precipitated by adding water. This was an oil of a different type from the others, having a high sp. gr. and a terpene-like odour. All the fractions except the last (which had the lowest viscosity) set above 0° and contained paraffin wax. The asphalt insoluble in glacial acetic acid was dissolved in carbon disulphide and decolorised by warming with dry bone-char. On removal of the solvent, a white crystalline wax was obtained, m.p. 67—75°. The remaining liquid contained a viscous, brown, fluorescent liquid. For comparison, crude oil was dissolved in ether, and successive quantities of alcohol were added to give fractions of diminishing sp. gr. A final fraction was precipitated by the addition of water. A study of the constituents of the oils soluble and insoluble in glacial acetic acid shows that the acid-soluble portion contains the lightest components and heaviest oils but no wax, whilst the acid-insoluble oils are of intermediate density and contain all the wax other than that present in the asphaltic portion.

H. S. GARLICK.

Action of sodium-potassium alloy on petroleum. J. B. CONANT and A. H. BLATT (J. Amer. Chem. Soc., 1928, 50, 542—550).—When treated with sodium-potassium alloy in dry ether at the ordinary temperature, certain crude petroleum yield intensely coloured metallic derivatives, which are converted by carbon dioxide into a mixture of salts of carboxylic acids. The following acids, obtained in this manner from the oils mentioned, are described: Pennsylvania crude, m.p. 90—100°, decomp. about 150°; Mid-continent crude, m.p. 120—130°, decomp. about 170°; Mid-continent cracking-coil residue, m.p. 140—150°, decomp. about 185°; California crude, m.p. 125—130°, decomp. about 150°; Columbia crude, m.p. 110—120°, decomp. about 140°. The acids appear to be dibasic, and the first-named acid yielded an imide. They also appear to contain one aromatic nucleus for each carboxyl group. Treatment of the Pennsylvania oil, after complete reaction with sodium-potassium at the ordinary temperature, with the alloy at 250° in presence of carbon dioxide, affords alkali salts of acids, together with a neutral, amorphous product. F. G. WILLSON.

Spontaneous decomposition reactions of berginisation: a reaction common to various petroleum hydrocarbons. E. PYHÄLÄ (Petroleum, 1928, 24, 308—314).—In connexion with attempts to lower the f.p. of still residues from Baku petroleum, a study was made of the behaviour of such substances in a cracking still of special design. A residue from Grosny petroleum was heated slowly to 450°, when a sudden rapid distillation and gas evolution occurred; 35% of the oil was recovered in this distillate, and by raising the temperature above 465° no further product was obtained. Three further experiments were carried out with Tscheleken petroleum, and it was found that reaction could first occur at about 380—400°, but that a second decomposition took place at 410—425° and produced about twice as much distillate as the first. Continued heating, up to 550° produced only negligible quantities of distillate, but the rate of the initial temperature rise materially affected the proportions of benzene, kerosene, etc. formed. 71% of the paraffin and 90% of the asphalt were found to have reacted without giving pitch or coke. Two experiments with an oil from Surachany, containing appreciable amounts of lubricating oil, showed that similar reactions could take place, and high percentages of paraffins and asphalts were decomposed. On account of the general occurrence of this phenomenon and the similarity between the temperatures required and those employed in the Bergius process, it is suggested that reactions of this type are always essentially concerned in the berginisation of coals. R. H. GRIFFITH.

Sulphuric acid test for mineral oils. K. TYPKE (Petroleum, 1928, 24, 314).—It has been proposed to test transformer and similar oils by their behaviour with strong sulphuric acid, the better samples showing less reaction, but the value of this method is now questioned. Apart from lack of information as to the true significance of the test, it is suggested that the presence of limited quantities of substances which are removed by the acid is desirable in order to increase the resistance of the oil to oxidation. R. H. GRIFFITH.

[Physiological] effects of "ethyl gasoline" and its combustion products. R. R. SAYERS, A. C. FIELDNER, W. P. YANT, and B. G. H. THOMAS (U.S. Bur. Mines, 1927, 447 pp.).—Animals exposed for eight months to the combustion products of "ethyl gasoline" showed no distinct storage of lead or symptoms of lead poisoning. The threshold concentration for lead dust is $0.14\text{--}1.4\text{ mg./m.}^3$. Vapour concentrations of $0.3\text{--}1\%$ (but not of $0.01\text{--}0.1\%$) caused storage of lead and symptoms of poisoning. Skin absorption experiments were also conducted. CHEMICAL ABSTRACTS.

Free-burning carbonised fuels for the open fire. H. GREGER (J. Fuel Soc. Japan, 1927, 6, 102—110, and J. Akita Min. Col., 1927, No. 3).—See B., 1927, 864.

Graphitic oxide. BROWN and STOREY, also BROWN.—See XI.

PATENTS.

[Sub-heated] coke ovens. N.V. SILICA EN OVENBOUW MIJ. (B.P. 270,765, 9.5.27. Ger., 8.5.26).—To prevent dirt falling into the controlling devices of the gas supply of vertical-flued ovens the gas is fed from a horizontal pipe into the vertical pipe which has at its lower end a removable plug forming a dust box. In the refractory material at the upper end is another duct closed by a plug which permits of inspection from the top to the bottom of the gas-supply ducts and the process of combustion, and cleaning can be carried out without interruption of the working of the ovens.

A. C. MONKHOUSE.

Heating of pulverised fuel. H. A. PROCTOR (B.P. 283,388, 30.4.27).—Pulverised fuel is distilled in horizontal chambers arranged one above the other and communicating with each other at the ends to form a continuous conduit. The fuel is conveyed by rotating paddles along the distillation chambers, which are arranged on one or both sides of a combustion chamber, and are provided with water-, steam-, or air-circulating pipes for regulating the temperatures therein.

A. B. MANNING.

Apparatus for the dry-cooling of coke. SOC. ANON. DES FOURS À COKE SEMET-SOLVAY & PIETTE (B.P. 282,286, 30.5.27. Belg., 5.5.27).—The coke is discharged from the oven into a chamber in which the walls of the coke receptacle are arranged to give a maximum exposed surface of coke. The boiler tubes are situated above and parallel with the coke surfaces. In the lower part of the chamber is a perforated bell for distributing the circulating gases from the fan through the coke, and after imparting their heat to the boiler tubes the gases pass through a chamber for dust removal.

A. C. MONKHOUSE.

Preparation of solid fuel mixtures. J. M. W. KITCHEN (B.P. 282,118, 15.6.26).—Coke or hard coal of relatively large size is mixed with soft bituminous slack, and for fusible coals anthracite dust is also added. A smokeless fuel is obtained by carbonising the mixture in a gas retort or travelling grate heated by the combustion of the carbonisation gases. [Stat. ref. to B.P. 217,236, 18,156 of 1895, and 4487 of 1887.]

A. C. MONKHOUSE.

Production of semi-coke briquettes from bituminous coal. KOKS- U. HALBKOKS-BRIKETTERUNGS-GES.M.B.H. (B.P. 279,020, 9.3.27. Ger., 14.10.26).—Briquettes are made by mixing fine bituminous coal with a binder consisting of a solution of one or more water soluble, preferably inorganic, substances which do not soften on heating, e.g., a mixture of sulphite waste liquor or cellulose pitch and clay, or coal dust emulsified with sulphite waste liquor. The waste liquor can be replaced by size, water-glass, etc. The briquettes are then dried and carbonised in a shaft furnace. A. C. MONKHOUSE.

Apparatus for carbonising fuels, particularly coal. R. TORMIN (B.P. 270,277, 19.4.27. Ger., 30.4.26).—Coal is carbonised in the form of briquettes in an apparatus consisting of a number of flat boxes with transverse partitions and corrugated bottoms which, when placed together, form hollow spaces joined together in rows and having the rhombic shape of the desired briquettes. When the juxtaposed boxes are placed upright, these spaces can readily be filled up with coal dust, since the inclined walls of the spaces are steeper than the angle of cohesion of ground coal. After carbonisation in a suitable retort the briquettes can be simultaneously discharged from the apparatus.

A. B. MANNING.

Carbonisation of briquettes and carbonisation plants therefor. MIDLAND COAL PRODUCTS, LTD., and C. INGMAN (B.P. 282,511, 5.10.26).—In the manufacture of briquettes, carbonisation is carried out in a primary retort under normal conditions, and the gas evolved is used in the heating of steam superheaters. The steam is employed for the carbonisation in secondary retorts either by itself or with a small proportion of air. A rich gas and higher yields of coked briquettes and oil are obtained than in the prior process (cf. B.P. 242,783; B., 1926, 37).

A. C. MONKHOUSE.

Distillation of carbonaceous substances. W. E. EVANS. FROM CARLSHÜTTE A.-G. F. EISENGIESSEREI U. MASCHINENBAU (B.P. 283,259, 2.10.26).—Coal, or similar fuel, is carbonised in a vertical retort by the transverse passage through it of a heating medium such as flue gases or steam. The retort is divided into zones by horizontal partitions. The heating medium passes first through the coke in the cooling zone, and then, via a heat exchanger arranged in the outlet chamber of the distillation zone, through the drying zone, or, after having heat supplied to it, is used directly for the distillation. While the heating medium passes through the heater, flue gases from the latter may be added to it if desired.

A. B. MANNING.

Distillation of solid fuels at low temperatures. COMP. DES MINES DE BRUAY (B.P. 272,522 and 282,340, [A, B] 9.6.27. Fr., [A] 14.6.26, [B] 18.12.26).—(A) Briquettes of pulverised coal or other fuel are imbedded in a mass of powdered material, e.g., silica, magnesia, kieselguhr, or metal filings, and carbonised at a low temperature with a carefully regulated rate of heating. A strong coke is produced which burns without smoke. (B) In a modification of the above process the imbedding material itself consists of carbonaceous material, e.g., dust from coal schist or roof coal, waste peat, sawdust,

vegetable waste, etc. The fuel also before briquetting is mixed with powdered charcoal. A. B. MANNING.

Recovery of liquid products from coal. H. NOVÁK and A. BROD (B.P. 283,269, 5.10.26).—Coal or brown coal is heated in an autoclave with an aromatic hydrocarbon boiling above 230°, the temperature being raised gradually from 200° to 500° in such a way that first the low-boiling products and then the high-boiling aromatic hydrocarbons are distilled off and condensed; the residual oils are then cracked above 380° and finally coked at 500°. Liquid homologues of naphthalene, fractions of anthracite or generator tar, or mixtures of these with solid aromatic hydrocarbons form suitable high-boiling liquids for the process. Considerably increased yields of oils are obtained, whilst the pressures necessary are comparatively low (25–30 atm.). A. B. MANNING.

Manufacture of activated charcoal. E. URBAIN (B.P. 257,269, 17.8.26. Fr., 22.8.25).—Carbonaceous materials are heated together with compounds containing oxygen, *e.g.*, metallic or metalloid oxides or hydroxides, at 800–1100°, without the introduction of gases. The oxides or hydroxides, which must be capable of reduction to metal or metalloid under the conditions specified, effect the removal of hydrogenated compounds from the material without reacting substantially with the charcoal. Substances other than oxides or hydroxides, *e.g.*, zinc chloride, which, however, yield oxides when heated to the temperatures used, *e.g.*, by interaction with the moisture present, can be used in the process. If the activating agent is highly volatile the impregnation and heating may be repeated.

A. B. MANNING.

Production of carbon electrodes. I. SZARVASY (B.P. 284,818, 6.12.26).—The mixture of carbon and tar-like products formed by decomposing gaseous hydrocarbons at high temperatures is coked, preferably in an atmosphere containing hydrogen and methane or other hydrocarbons, at a temperature not exceeding 1700°, and the product worked up into electrodes by being ground, mixed with a binder, moulded, and calcined. The hard carbon deposited on the walls of the decomposition vessel may be mixed in with the coked product or may be worked up separately into electrodes.

A. B. MANNING.

Gas generators. C. W. ANDREWS and W. B. CHAPMAN (B.P. 284,805, 22.11.26).—In a gas generator provided with a beam which rotates over the grate for the purpose of discharging the ash and agitating the fuel bed the ash is collected in an annular trough attached to the beam, the rate of removal of ash from this trough being controlled by an adjustable plough projecting thereinto. A pressure chamber beneath the grate serves to receive the air, steam, etc. fed to the generator. The inner faces of the side walls are formed with ridges which co-operate with the ends of the beam to grind up any large clinkers in the ash as it passes to the trough. The beam and the driving ring to which it is attached may be hollow, in which case means are provided for the passage of cooling water through them.

A. B. MANNING.

Working of water-gas and double-gas producers. H. STRACHE (B.P. 269,936, 23.4.27. Austr., 24.4.26).—In operating a gas producer with an alternate "blow"

and "run," instead of removing coke from the producer and thereby increasing the calorific value of the gas by augmenting the proportion of distillation gases therein, the coke is gasified in the producer by prolonging the "blow," the carbon monoxide produced being used for heating or power purposes. The higher temperatures attained increase the efficiency of the process during the "run."

A. B. MANNING.

Production of gaseous fuels. SYNTHETIC AMMONIA & NITRATES, LTD., and K. GORDON (B.P. 282,141, 11.9.26).—Water-gas is produced from finely-divided, high-grade, solid fuel and steam by blowing the mixture, with or without oxygen or air, through an externally heated reaction vessel consisting of an alloy-steel tube or tubes made from "Staybrite," "Era A.T.V.," or "V2A." Using a tube 30 ft. long and $\frac{1}{2}$ in. diam. heated at about 1000°, coke and steam were converted into a water-gas containing 51% H₂, 48% CO, 1% CO₂; the time of contact was 1 sec.

A. C. MONKHOUSE.

Manufacture of producer gas. C. W. ANDREWS (B.P. 260,628, 1.11.26. U.S., 31.10.25).—Producer gas containing carbon monoxide as its main combustible constituent is made by passing air, mixed with steam or metallurgical gases, alternately up and down through a deep fuel bed in such a manner that the upper layer and the lower layer (and ash) of the fuel serve alternately as regenerators; or the hot producer gas may be passed through a separate regenerator, which is used to heat the air for the succeeding "blow."

A. B. MANNING.

Production of volatile hydrocarbons such as gasoline, benzene, or other hydrocarbon oils contained in natural gas, coal gas, water-gas, air, or other gaseous mixtures. M. OKOCHI (B.P. 282,541, 12.11.26).—The gaseous mixture is compressed, cooled, and then suddenly expanded into a tower packed with absorbing material. The dried gases leave the tower and are used for heating purposes. The temperature in the tower is controlled by means of the admission of uncompressed gas. When the absorption material becomes saturated the volatile materials are expelled by a current of hot gas and condensed; the tower is then cooled ready for subsequent absorptions.

A. C. MONKHOUSE.

Distillation of tar and other viscous substances, and apparatus therefor. J. F. CARMICHAEL and J. F. CARMICHAEL & Co., LTD. (B.P. 282,525, 28.10.26).—The material to be distilled is distributed on to an annular or spiral coil or series of coils situated in a chamber. The coils are heated internally and the material flows by gravity over the surface of the coils and is withdrawn at the bottom of the chamber. The vapours evolved pass from the top of the chamber to a condenser.

A. C. MONKHOUSE.

Production of a composition of matter [from pitch]. L. BOLGAR (B.P. 277,291, 2.5.27. Hungary, 13.9.26).—Molten coal-tar pitch or petrol pitch (with, if desired, natural asphalt) is mixed with not more than 5% by wt. of sulphuric acid (*d* 1.53), and heated to 200–250°; the liquid product, after being mixed with filling material, *e.g.*, sand, stone-meal, clay, etc., is cast into the desired shape. H. ROYAL-DAWSON.

Production of gasoline from natural gases. E. B. BIRD (U.S.P. 1,656,813, 17.1.28. Appl., 19.3.27).—Natural gas is circulated at 400–500° over a catalyst comprising a mixture of copper, nickel, gold, platinum, and palladium to form liquid hydrocarbons which are condensed and separated. T. S. WHEELER.

Breaking of petroleum emulsions. M. DE GROOTE and W. C. ADAMS, Assrs. to W. S. BARNICKEL & Co. (U.S.P. 1,656,622—3, 17.1.28. Appl., 11.8.26).—(A) A tar acid oil is treated with sufficient sulphuric acid to sulphonate only the phenols present, the product is mixed with oleic acid which has been treated with sulphuric acid, and the mixture is treated with this acid to give a product containing a Twitchell reagent. (B) A mixture of a phenol, *e.g.*, β -naphthol, and a fatty acid, *e.g.*, oleic acid, is sulphonated to give a Twitchell reagent. Both products induce rapid separation of petroleum emulsions. T. S. WHEELER.

Apparatus for the conversion of hydrocarbon oils. E. T. HESSLE and W. LÖLGEMANN (U.S.P. 1,658,985, 14.2.28. Appl., 20.3.25).—Oil and gas are introduced into a reaction kettle through opposed nozzles, whereby a spray of oil and gas is produced and brought into contact with a catalyst. C. O. HARVEY.

Control of pyrolytic conversion [of hydrocarbon oils]. J. R. CARRINGER, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,656,724, 17.1.28. Appl., 28.5.23).—The oil is heated to a high temperature and passed to a chamber in which cracking is allowed to progress at a slightly lower temperature. To control this lower temperature a desired portion of the oil vapour passing to the chamber is led through a coil cooled by air. T. S. WHEELER.

Cracking of hydrocarbon oils. C. P. DUBBS (B.P. 284,507, 29.4.27).—In a process involving the continuous cracking of hydrocarbon oils by passage through a heating coil (at about 427–482°) to a reaction zone and dephlegmator, the proposed introduction of an incompressible gas to the reaction zone (*cf.* B.P. 249,604; B., 1926, 525) is modified by subjecting the gas to a separate external heating operation (at about 644–816°) and making use of gas other than that produced during the cracking operation. C. O. HARVEY.

Cracking of [hydrocarbon] oil. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,659,398, 14.2.28. Appl., 11.1.26).—Oil is cracked continuously under its self-generated super-atmospheric vapour pressure by passage through a cracking coil to the large end of a conical expansion chamber, wherein the rate of flow of the evolved vapours (and the rate of conversion) is gradually increased as they pass axially along the cone. The vapours, passing out from the apex of the conical chamber, pass through a dephlegmating column, and the condensate therefrom is combined with the feed oil, the remaining vapours being condensed and collected. C. O. HARVEY.

Apparatus for treatment of hydrocarbons. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,659,397, 14.2.28. Appl., 23.7.20. Renewed 2.9.26).—A horizontal cylindrical still and a heating element contained therein are arranged

to rotate in opposite directions. The still is fitted with an inlet for oil, and with outlets for residuum and vapours, the last being refluxed and the resulting condensate being returned to the still. C. O. HARVEY.

Refinery power plant [for hydrocarbon oils]. M. J. TRUMBLE (U.S.P. 1,658,910, 14.2.28. Appl., 1.12.22).—Hot oil is delivered to a regulating device and the expansion of the evolved vapours is used to operate a turbine. A substantially constant pressure is maintained by adding vapour to the hydrocarbon vapours from the oil. C. O. HARVEY.

Burning of oil fuel. J. E. HACKFORD (B.P. 284,777, 4.11.26, 8.1.27, and 18.2.27).—Oil fuel is burnt without atomisation by passing a steady stream of air over a concave surface (a cup or trough) which is covered with a thin film of the oil, the oil being fed to the surface at such a rate as to maintain this film. Means are provided for varying the heat intensity of the flame by adjusting the direction etc. of the air current. In its simplest form the apparatus consists of a cup into which the oil is allowed to drip from a gravity feed. C. O. HARVEY.

Manufacture of refined lubricating and like oils. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ (B.P. 263,167, 15.12.26. Holl., 15.12.25).—Oils of low "cloud test" (*i.e.*, comparatively free from paraffin wax) are produced by extraction at a low temperature with liquid sulphur dioxide, which yields an extract free from wax and a residue saturated in sulphur dioxide, from which the wax may be removed by filtration. The sulphur dioxide reduces the viscosity of the mixture to be filtered and the solubility of the wax in the oil; its removal by evaporation is easily effected, and the pressure developed may be used to force the oil through the filters etc. The acid extract containing aromatic hydrocarbons etc. may, if desired, be recombined with the de-waxed residue. C. O. HARVEY.

Preparation of artificial petroleum and de-colorising carbonaceous substances from rubber scrap or vulcanised rubber waste. H. NISHIDA and K. SHIMADA (B.P. 282,565, 22.1.27).—Rubber waste or the oil obtained from its distillation is mixed with a natural bleaching earth, and distilled in an iron retort to 350°. The distillate is fractionated and washed with sulphuric acid and caustic soda. The residue is in the form of a black powder. A. C. MONKHOUSE.

Manufacture of agglomerated carbonaceous material. E. URBAIN, Assr. to URBAIN CORP. (U.S.P. 1,659,931, 21.2.28. Appl., 29.4.24. Fr., 25.6.23. Renewed 20.7.27).—See B.P. 218,242; B., 1925, 435.

Gas-purification process and apparatus. E. H. BIRD, Assr. to KOPPERS Co. (U.S.P. 1,660,741, 28.2.28. Appl., 24.11.22).—See B.P. 207,488; B., 1924, 459.

Production of lighter hydrocarbons. E. S. ANDREWS. From B. ORMONT ASSOCIATES, INC. (B.P. 285,199, 11.12.26).—See U.S.P. 1,608,664; B., 1927, 163.

Dehydration of alcohol for carburants. E. RICARD and H. GUINOT, Assrs. to SOC. RICARD, ALLENET, & C^{ie}. (U.S.P. 1,659,958, 21.2.28. Appl., 27.9.23. Belg., 30.9.22).—See B.P. 204,697; B., 1924, 899.

Smoke washers. W. W. ROBINSON (B.P. 284,919, 7.6.27).

Regeneration of lubricating oils contaminated in thermal engines. L. MIRLESSE and A. I. KOUSNETZOFF (B.P. 269,148, 14.3.27. Fr., 7.4.26).

Viscosity tester for oils (U.S.P. 1,659,534).—See I. Deodorisation of isopropyl alcohol (U.S.P. 1,657,505).—See III.

III.—ORGANIC INTERMEDIATES.

Catalytic chlorination of acetic acid to chloroacetic acid. H. BRÜCKNER (Z. angew. Chem., 1928, 41, 226—229).—The velocity of chlorination of acetic acid in presence of iodine, phosphorus, or sulphur increases with the amount of catalyst used, but not in direct proportion. Of the three catalysts iodine gives the best results, but more effective chlorination is obtained with mixtures of three catalysts, chlorination in presence of a mixture of iodine, phosphorus, and phosphorus pentachloride being more rapid than in presence of sulphur, phosphorus pentachloride, and phosphorus. Binary mixtures of catalysts are less effective than the ternary mixtures. Acetic anhydride is chlorinated in the absence of a catalyst, and in the chlorination of acetic acid the presence of chloroacetyl chloride and the mixed anhydride of acetic and chloroacetic acid has been established. The suggested mechanism of the chlorination is represented by a series of equations. Sulphur and phosphorus appear to act by accelerating the formation of acetyl chloride, and iodine by the dispersion of the chlorine through the reaction mixture in consequence of the formation of iodine chloride and iodine trichloride. R. BRIGHTMAN.

Determination of alcohol in solutions containing acetone. J. M. MACOUN (J.S.C.I., 1928, 47, 43—44 r).—About 50 c.c. of the sample are weighed into a 500 c.c. Kjeldahl flask, benzaldehyde is added in excess (3 c.c. for every 1 c.c. of acetone supposed to be present) and 1 g. of solid potassium hydroxide to 10 c.c. of aqueous liquid, and the mixture digested at its b.p. for 30 min., after which 100 c.c. of water are added to the cooled liquid and the mixture is distilled, using a 500 c.c. separating funnel as receiver. The distillate consists of alcohol, water, and excess of benzaldehyde. To not less than 100 c.c. of distillate 50 c.c. of saturated salt solution and enough solid sodium chloride to saturate the whole are added, followed by 75 c.c. of light petroleum, and the benzaldehyde extracted by Thorpe and Holmes' method (B., 1903, 232). The ethereal layer is washed with two successive portions (25 c.c.) of salt solution, and the extract and washings are distilled into a tared flask fitted with a glass stopper. The weight of the distillate (which should not be less than 100 c.c.) and its sp. gr. are ascertained, and from this the percentage of alcohol by wt. is calculated. If the n_D test for indicating methyl, ethyl, or isopropyl alcohol is to be relied on, the trace of benzaldehyde in the distillate must be eliminated, either by an elaboration of the above method, or less satisfactorily by the action of phenylhydrazine hydrochloride on the benzaldehyde before final distillation. D. G. HEWER.

Nephelometric method for determining benzene in alcohol. L. DESVERGNES (Ann. Chim. analyt., 1928,

[ii], 10, 6—11).—The approximate solubility of benzene in water and in alcohol-water mixtures has been determined for several temperatures. The turbidity produced when alcohol-water solutions of benzene are cooled to -15° may be used to determine the percentage of benzene by comparison with the clouds formed by solutions containing known amounts of the hydrocarbon. Should no cloud be produced, distillation gives an initial benzene-rich fraction, in which the benzene concentration may be determined as described. L. M. CLARK.

Determination of anthracene and anthraquinone. H. PIRAK (Z. angew. Chem., 1928, 41, 231—233).—In the original Höchst method the results obtained are too low on account of over-oxidation; the modified method of Rhodes and Sielisch (B., 1926, 941) gives low results through incomplete oxidation, and a slight excess of chromic oxide solution over the amount indicated by them is necessary for accurate results. The time required for the determination may be shortened by diluting the hot sulphuric acid solution of anthraquinone with boiling sulphuric acid. Loss by sublimation may be high at 110° in the original Höchst test (cf. Rhodes and Sielisch, *loc. cit.*) but is reduced by use of the crucibles advocated by Jacobsohn (B., 1926, 734). A lower temperature is, however, preferred. In the procedure recommended, anthracene (1 g.) is dissolved and oxidised as in Sielisch's method, a further 1 c.c. of the chromic acid solution being added when the solution is definitely brown. After cooling and dilution with 400 c.c. of ice-cold water, the anthraquinone is collected on a Jena glass filter-crucible, washed as in Jacobsohn's method, and dried at $95-100^\circ$. The contents are then transferred to a glass beaker and heated with 10 c.c. of sulphuric acid monohydrate at 105° (boiling brine-bath) for 20 min.; acetic acid (25 c.c.), free from traces of reducing impurities and previously heated to 100° , is added. On cooling, the anthraquinone separates as a white lump at the bottom of the beaker. When the contents reach room temperature, the beaker is placed in ice for 20 min. and 200 c.c. of cold water are then added, rise of temperature being avoided by occasionally cooling in ice. The anthraquinone is collected on the original filter, washed as before, and dried at $95-100^\circ$ to constant weight, after which it is sublimed at $180-200^\circ$ (1 hr.) in a drying oven. The door of the oven is not completely closed, and the stopper carrying the thermometer is notched to allow of a slight air-current. The determination is complete in 4—5 hrs., and gives satisfactory results with technically pure anthracene and anthraquinone, and with crude anthraquinone. With mixtures containing less than 50% of anthraquinone the method gives low results for anthraquinone, and consequently high values for anthracene. R. BRIGHTMAN.

n-Butyl alcohol, acetone, and methyl alcohol from maize. TROPSCH.—See XVIII.

PATENTS.

Method of retarding or preventing the corrosion of metal containers containing alcohol. U.S. INDUSTRIAL ALCOHOL Co., Assecs. of W. T. SCHREIBER (B.P. 269,135, 1.12.26. U.S., 6.4.26).—Containers (especially those made of tin plate,terne plate, or iron) for commer-

cial alcohols, especially commercial or denatured ethyl alcohol, may be protected against corrosion by dissolving in 100 c.c. of the alcohol 0.01–0.25 g. of soap (*e.g.*, alkali soaps of palmitic, stearic, or oleic acid, etc., or rosin soap) or soap-forming materials (such as the higher fatty acids).
B. FULLMAN.

Deodorisation of isopropyl alcohol. C. S. HUDSON, ASSR. to STANDARD DEVELOPMENT Co. (U.S.P. 1,657,505, 31.1.28. Appl., 28.6.21).—Calcium hypochlorite and hydrochloric acid are added to isopropyl alcohol, prepared from hydrocarbon material, and the mixture is agitated.
H. ROYAL-DAWSON.

Production of acetic anhydride. C. RUZICKA (B.P. 283,781, 27.5.27).—Sulphur trioxide fumes, produced by direct distillation of the trioxide or by oxidation of sulphur dioxide with air or oxygen (preferably activated or ozonised) in the presence of a catalyst, are conducted into cooled acetic acid, glacial or otherwise, with agitation. The product may be used directly (*e.g.*, in the acetylation of wood cellulose), or distilled for the separation of acetic anhydride.
B. FULLMAN.

Manufacture of acetic acid. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 284,143, 7.7.27. Ger., 7.4.27).—A small quantity of calcium acetate is dissolved in acetic acid and treated with sulphuric acid. Thereafter acetic acid may be continuously produced by dissolving calcium acetate in the paste of acetic acid and calcium sulphate and adding sulphuric acid. Excess of the latter is avoided.
B. FULLMAN.

Manufacture of [crystalline] urea. I. G. FARBEININD. A.-G. (B.P. 266,378, 19.2.27. Ger., 19.2.26).—Needle-shaped crystals of urea are produced by exposing drops of a solution containing between 50% and 90% by wt. to slow cooling in a current of air or other gas.
B. FULLMAN.

Manufacture of acylamides. R. B. TRUSLER, ASSR. to ROESSLER & HASSLACHER CHEMICAL Co. (U.S.P. 1,656,252, 17.1.28. Appl., 27.12.26).—Primary or secondary amines, which are less basic than ammonia, and react with acid anhydrides to form amides, are heated with amide hydrogen sulphates to yield the corresponding alkylated amide and ammonium hydrogen sulphate. *E.g.*, aniline and formamide hydrogen sulphate give formanilide.
T. S. WHEELER.

Manufacture of aromatic hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 258,608, 17.9.26. Ger., 12.10.25).—Aromatic (especially benzenoid) hydrocarbons are obtained on passing methane, or gases containing it, at 500–800° and under 50–1000 atm. pressure, over catalysts. The latter may be carbonates or other salts of the alkaline-earth metals, including magnesium and beryllium; the hydroxides of the last two, or their oxides (especially beryllium oxide); compounds of selenium, tellurium, or thallium; or active silica or charcoal; alone or mixed.
B. FULLMAN.

Production of ester mixtures. W. CLAASEN (U.S.P. 1,656,883, 17.1.28. Appl., 7.3.27. Austr., 29.1.27).—The process of B.P. 250,910 (B., 1926, 769) is applied to cyclohexanol.
T. S. WHEELER.

Preparation of leuco-quinizarin. I. GUBELMANN and H. R. LEE, ASSRS. to NEWPORT Co. (U.S.P. 1,655,462,

10.1.28. Appl., 25.1.26).—Chloroquinizarin in acid solution is treated with aluminium powder at room temperature, and the reaction mixture is poured into water.

T. S. WHEELER.

Production of 4-nitro-2-aminophenoxy-ethanol or -propandiol [4-nitro-2-aminophenyl β -hydroxyethyl or $\beta\gamma$ -dihydroxypropyl ether]. CHEM. WORKS FORMERLY SANDOZ [CHEM. FABR. VORM. SANDOZ] (B.P. 263,191, 18.12.26. Ger., 19.12.25).—2:4-Dinitrophenyl β -hydroxyethyl and β -dihydroxypropyl ethers are reduced, without hydrolysis of the ether group, to the nitro-amino-ethers by aqueous sodium sulphides, especially by the tetrasulphide, at 60°. 4-Nitro-2-aminophenyl β -hydroxyethyl ether, m.p. 114°, and the $\beta\gamma$ -dihydroxypropyl ether, m.p. 111–112°, are described.

C. HOLLINS.

Concentration of acetic acid. J. DUCLAUX, ASSR. to FABR. DE SOIE ARTIFICIELLE DE TUBIZE SOC. ANON. (U.S.P. 1,660,418, 28.2.28. Appl., 31.3.24. Ger., 4.4.23).—See B.P. 213,914; B., 1925, 227.

Carbon disulphide (B.P. 282,049).—See VII.

IV.—DYESTUFFS.

Photographic properties of cyanine dyes. BLOCH and HAMER. Dyes and fogging reactions. LÜPPO-CRAMER.—See XXI.

PATENTS.

Basic dye. J. KERN, ASSR. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,660,167, 21.2.28. Appl., 26.12.22).—A dye suitable for textile printing is obtained by heating to boiling a solution of a basic dye and tannin.

A. R. POWELL.

Production of oil-soluble azo colouring matters. J. P. PENNY, ASSR. to NAT. ANILINE & CHEMICAL Co., INC. (U.S.P. 1,656,322, 17.1.28. Appl., 2.3.26).—An excess of a diazotised arylamine is added to a phenol, and, after coupling is complete, the mixture is heated to decompose the excess of diazo compound. The mixture of azo dye and the decomposition products of the diazo compound is more soluble in hydrocarbons than the pure dye.

T. S. WHEELER.

Manufacture of monobenzoyldiaminoanthraquinone compounds. W. H. PERKIN and H. M. BUNBURY, ASSRS. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,660,090, 28.2.28. Appl., 4.3.26. U.K., 8.7.25).—See B.P. 255,630; B., 1926, 817.

Manufacture of an azo dye [1-(*o*-chloro)phenyl-3-methyl-5-pyrazoloneazobenzene]. M. SCHMID, ASSR. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,660,097, 28.2.28. Appl., 19.7.26. Switz., 1.8.25).—See B.P. 256,205; B., 1927, 869.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Preservation of fishing nets by treatment with copper soaps and other substances. W. R. G. ATKINS (J. Marine Biol. Assoc., 1928, 15, 219–235).—For the preservation of cotton and hemp net copper oleate has no advantages over a mixed copper soap containing oleate. Details are given of tests of various preserving agents, and it is concluded that the best results are

obtained by the use of copper soap reinforced with anti-fouling paint or coal tar, petrol being better than paraffin as a diluent. After this treatment, nets, the life of which when untreated is from 2 to 3 months in aquarium water, are sound after 18 months. W. O. KERMACK.

Action of formaldehyde on wool. S. R. TROTMAN, E. R. TROTMAN, and J. BROWN (J. Soc. Dyers and Col., 1928, 44, 49—52).—The reaction between wool and formaldehyde is incomplete at ordinary temperatures, proceeds more rapidly with rise of temperature, and is complete in 3 hrs. at 75°, when the amount of combined formaldehyde is 0.7% on the weight of dry wool. The combination with wool under these conditions is more stable than that with leather. Formaldehyde has little protective action for wool in a soap scouring bath, a chlorinating bath, or in cold dilute solutions of sodium carbonate or hydroxide. The moisture content of air-dried paraformaldehyde wool is considerably less than that of untreated wool, intermediate values being found for the formaldehyde combination. No change occurs in the nitrogen content as a result of the treatment of wool with either formaldehyde or paraformaldehyde, but the former gives the greater resisting power against the action of cold sodium hydroxide solution. Formaldehyde wool takes up more sodium hydroxide from solution than untreated wool, is resistant to boiling for 1 hr. with 0.5% sulphuric acid, and has a lower shrinkage than untreated wool when washed normally. It has a greatly reduced affinity for neutral dyeing acid dyestuffs (though in the presence of acid normal dyeing is obtained), is resistant to bacterial action, and may be diazotised. These properties are attributed to the displacement of the peptide equilibrium in wool to the enol form, the latter being more resistant to decomposition by acids and alkalis than the keto form. At raised temperatures the enol form tends to revert to the keto form, and formaldehyde wool then behaves similarly to untreated wool. B. P. RIDGE.

Viscose. IX. G. KITA, R. TOMIHISA, K. AZAMI, and M. FUJIMOTO (J. Soc. Chem. Ind., Japan, 1927, 30, 198—204).—The action of solutions of sodium chloride, zinc sulphate, and copper sulphate, respectively, on viscose has been studied. The alkali is not removed by washing with alcohol the xanthate that has been purified with dilute acetic acid and sodium chloride solution. The purified xanthate is gradually decomposed by drying, and a part of the alkali thus set free seems to enter into combination with the carbon disulphide, producing a compound which is neutral to phenolphthalein. Sodium xanthate is much more stable than ammonium xanthate. When zinc sulphate was added to viscose, 75% of the sodium xanthate was found to be substituted by the zinc salt, a white precipitate of the latter being produced which is difficultly soluble in water but readily soluble in alkali. The amount of carbon disulphide combined as xanthate is decreased by drying, but not by treatment with zinc sulphate. When copper sulphate solution is mixed with purified xanthate, a yellowish-brown precipitate of copper xanthate is obtained, which is stable towards dilute sulphuric acid and hydrochloric acid at ordinary temperatures, but is decomposed at 70°. The amount of copper in copper xanthate was about

75% of the amount of carbon disulphide combined in the form of xanthate. Y. TOMODA.

Ripening of viscose. J. FRENKEL (Cellulosechem., 1928, 9, 25—26).—The original hypothesis of Cross and Bevan that the cellulose molecule of viscose undergoes polymerisation during the ripening process could not be upheld, and it was replaced by the conception of a progressive hydrolysis of the xanthogenic ester in aqueous alkaline solution which corresponds with the analytical results. There remains the question whether the colloid remaining in dispersion in the fully ripened viscose, represented analytically by the so-called "C 24 xanthate," is a chemically homogeneous ester of lower degree of esterification or is a mixture of the original "C 12 xanthate" with free cellulose hydrate resulting from the hydrolysis of a portion of it. The author considers that the weight of evidence favours the latter conception. This is based on the possibility of washing out with water the soluble portion of a ripened viscose coagulated with ammonium chloride, and leaving a residue of pure cellulose hydrate. Further, it is possible to disperse by the Plauson colloid mill 3 pts. of freshly-made viscose with hydrated cellulose regenerated by the action of acid on 1 pt. of the same viscose, and to obtain with the addition of caustic soda a ripened viscose indistinguishable from one ripened in the usual way.

J. F. BRIGGS.

Cellulose xanthate and viscose. I. Reactions involved in the ripening of viscose. K. ATSUKI. II. Changes of the colloidal nature of viscose with the concentration of free alkali. K. ATSUKI, I. OKAMURA, and T. MATSUDA (J. Fac. Eng., Tokyo, 1927, 17, 135—144, 145—151).—I. During the course of the ripening of viscose, the viscosity and surface tension decrease, reach a minimum, and then increase, whilst the amount of 25% ammonium chloride solution necessary for coagulation decreases rapidly at first, and then very slowly, finally becoming nearly constant. The results indicate that the ripening takes place in two stages; dehydration and disintegration of the disperse phase (a concentrated solution of sodium cellulose xanthate) first occur, until the point of minimum viscosity and surface tension is reached, when the solution is suitable for spinning, after which coagulation of the excessively dehydrated particles commences. Determinations of the combined sodium and sulphur present at various stages of the ripening show that no appreciable hydrolysis occurs until the colloidal dispersion reaches its maximum, but it becomes more pronounced when coagulation sets in, the colloidal instability apparently giving rise to chemical instability.

II. From observations of the changes in specific volume of the disperse phase and the surface tension of viscose with increase of the concentration of free alkali present during ripening, it is deduced that when the alkali concentration is about 6—8%, the disperse phase (a concentrated alkaline solution of sodium cellulose xanthate) shows the highest degree of dispersion, and the colloidal system is most stable. With a lower concentration of free alkali there is an increased tendency to hydrolysis, and the disperse phase contains a larger proportion of water of hydration, whilst with a higher

concentration the particles are excessively dehydrated and tend to coagulate. W. J. POWELL.

Manufacture of cellulose; alkalinity changes, and the action of sodium sulphide [in the cooking process]. C. KULLGREN (Ing. Vet. Akad., Stockholm, 1927, No. 65, 34 pp.).—The ordinary titrimetric method of studying the changes of alkalinity of the liquor during the cooking process gives results which are too low for the early stages and too high for the later stages of cooking. When sodium hydroxide alone is present an electrical conductivity method may be used, but when carbonate and sulphide are also present, as in black liquor, a reaction velocity method is more suitable. In the latter the reaction velocity is measured by means of the catalytic decomposition of diacetone alcohol by alkali into acetone, a unimolecular reaction in which the velocity is proportional to the hydroxyl-ion concentration. Measurements are carried out in a dilatometer and the constants obtained are approximately proportional to the sodium hydroxide content of the liquor. In cooking with sodium hydroxide alone a cessation or pronounced decrease in the attack of the wood occurs within the temperature range 130–150°, whilst above 160° considerable decomposition again takes place. When sulphide is also present such stagnation is negligible and the alkali content of the liquor decreases uniformly. This process for the manufacture of sulphate cellulose depends on the fact that, although the sulphide itself is a bad decomposition agent, it forms sulphur compounds (lignins containing sulphur) which are more easily attacked by sodium hydroxide than is a wood substance containing no sulphur, the attack taking place rapidly at 140–160°. B. P. RIDGE.

Behaviour of cellulose towards acids. P. E. ALTMANN (Chem.-Ztg., 1928, 52, 150–151).—A short résumé of existing knowledge concerning the qualitative behaviour of cellulose with various mineral and organic acids. A product similar to the artificial parchment prepared by steeping paper in 60% sulphuric acid and washing may be prepared without the use of acid by coating unsized paper with a solution containing 125 g. of sodium salicylate and 200 g. of glycerin in 1.25 litres of water. W. J. POWELL.

Cellulose esters. I. Depolymerisation and mechanical properties of the nitrates. H. OKADA (J. Cellulose Inst., Tokyo, 1927, 3, 285–296).—Having worked out a method for preparing regular nitrocellulose films under standardised conditions, the author has studied the relations between viscosity and mechanical properties. Various types of cellulose in the form of cotton, cotton celluloid tissue paper, bleached sulphite wood pulp, and sulphite pulps specially purified with lime and sodium sulphite were subjected to preliminary treatments calculated to induce depolymerisation while avoiding definite hydrolysis and oxidation. Treatments comprised heating at 145° and 125° with water, dilute alkali, and glycerol; treatment with sodium hydroxide under mercerising conditions; regeneration from alkali cellulose after various ageing periods; regeneration from viscose and in the form of viscose silk; treating nitrocellulose with water under pressure. These modified celluloses were nitrated under identical conditions, the

viscosities of the nitrated products were determined in 1% acetone solution, and films were made 0.10–0.12 mm. thick from the ether-alcohol solutions. Over a wide range of relative viscosities from 126 down to 2.5 the tenacity of the films was almost constant at 15 kg./mm.², and the crumpling resistance was rather less constant at about 200. The influence of depolymerisation of the cellulose, whilst very marked on the viscosity, had a nearly negligible degrading effect on the mechanical properties. Only at a very advanced stage of depolymerisation, with reduction of the viscosity figure below 2, was a very sudden breakdown of the mechanical resistance of the nitrocellulose film recorded, obviously brittle products being thereby produced.

J. F. BRIGGS.

Researches on wood chemistry. III. Composition of Tsuga and Momi. Y. UEDA and T. YOSHIDA (J. Cellulose Inst., Tokyo, 1927, 3, 297–298).—Air-dried samples of Japanese pulp woods *Tsuga* (*T. Sieboldii*) and *Momi* (*Abies firma*) on analysis gave: cellulose 48.71%, 45.24%; lignin 21.53%, 25.74%; furfuraldehyde (as pentosans) 4.69%, 6.26%, and resins (as benzene extract) 2.17%, 2.26%, respectively. These woods bear a close similarity in composition to the Kabafuto spruce, but showed an exceptional difficulty in the chlorination process of analysis due to the strong affinity between the cellulose and the lignin in these woods; this has an important significance in the pulping process. Owing to their compact structure these woods, especially *Tsuga*, are only suitable for pulping by chemical methods.

J. F. BRIGGS.

Silks and dyestuffs. HOUCK.—See VI. **Nitrocellulose solvents.** BROWN, also BRIDGMAN.—**Nitrocellulose thinners.** DAVIDSON and REID.—See XIII. **Hemicellulose-splitting enzyme.** LÜERS and VOLK-AMER.—See XVIII.

PATENTS.

Apparatus for the production of fibre from flax and hemp straw and the leaves of *Phormium tenax*, sisal, abacca, yucca, etc. J. C. W. STANLEY, ASSR. to OCEANIA INDUSTRIES, LTD. (U.S.P. 1,657,046, 24.1.28. Appl., 18.4.27).—Apparatus is described for the continuous treatment of textile raw material for the production of spinnable fibres, and comprises conveyors, washing and shaking screens, and tipping tanks for alkaline digestion, bleaching, and oiling operations. After treatment with alkali the washed fibres are conducted through a water flume provided with paddles between and below which are arranged perforated pipes for injecting air under pressure. The material is finally dried in a current of heated ozonised air.

D. J. NORMAN.

Production of sterilised and stable vegetable bristle or fibre. G. AVERSENG (B.P. 284,421, 10.11.26).—Vegetable bristles or fibres obtained from the leaves of palm trees are freed from soluble mineral salts, pectic matter, and fermentable juices by extraction with cold or hot water, optionally under pressure and in the presence or absence of alkalis, and are simultaneously or subsequently treated with a solution of an organic or mineral antiseptic or anticryptogamic agent such as copper sulphate, zinc sulphate, salicylic acid, etc. A

suitable solution contains 2% of copper sulphate on the weight of fibre.

D. J. NORMAN.

Treatment of fibre. A. F. RICHTER and F. A. AUGSBURY, Assrs. to STEBBINS ENGINEERING AND MANUF. CO. (U.S.P. 1,653,416, 20.12.27. Appl., 19.7.26).—The hot high- and low-pressure relief gases from the digester are passed into an accumulator tank, where they are absorbed under pressure by fresh acid liquor. The hot liquor thus obtained is used for the treatment of further quantities of raw material.

D. J. NORMAN.

Treatment of Spanish moss. R. STINSON (U.S.P. 1,656,842, 17.1.28. Appl., 10.6.24).—The moss is submerged in a solution of ferrous or ferric chloride or sulphate until the tannic acid present has reacted with the metal, and is then dried and the husk removed.

T. S. WHEELER.

Extraction of fatty matter from foams formed by the beating, froth-flotation, or other treatment of wool-washing waters. E. MERTENS (B.P. 273,642, 27.10.26. Belg., 1.7.26).—The foam is maintained at 80–90° in a digester, and the soaps, albuminous matter, etc. which collect with the water at the bottom of the digester are withdrawn. Fresh water is added and the operation repeated as many times as may be desirable. The digester is then closed and the temperature increased until a pressure of 2–3 atm. is reached. For a charge of 1000 kg. of foam, containing 20% of neutral fat, 10 hrs. at pressure is sufficient. 80% of the fat initially present is recovered in a homogeneous, anhydrous form. No chemicals are used at any stage of the process.

D. J. NORMAN.

Production of brocaded textiles. H. FLORY (U.S.P. 1,655,414, 10.1.28. Appl., 9.10.25).—Fabric having a cotton back and a pile face of wool is printed with a solution containing sodium hydroxide, sodium sulphate, and gum arabic, and the disintegrated pile fibres are removed from the treated portions.

T. S. WHEELER.

Impregnation or coating of fabrics, paper, or the like web material. E. WEINHEIM (B.P. 255,094, 10.7.26. Ger., 13.7.26).—The material is subjected to a constant and uniform tension both during impregnation and drying, suitable apparatus for which is described. [Stat. ref. B.P. 122,680—1, 4494 of 1915, and 26,180 of 1910.]

D. J. NORMAN.

Preparation of painting grounds for artists, decorators, and craftsmen. C. F. CROSS, VISCOSÉ DEVELOPMENT CO., LTD., and WINSOR & NEWTON, LTD. (B.P. 284,363, 26.8.26).—Cellulosic textile material is filled and coated with cellulose partly as fibrous cellulose and partly as regenerated cellulose. Thus, unsized paper is attached to a fibrous base either during the manufacture of the paper or subsequently by the use of viscose solution as an adhesive, and this composite material is impregnated with viscose solution containing, e.g., 4% of cellulose. The impregnated sheet is kept until reversion of the viscose is complete, after which it is washed free from soluble salts. Alternatively, paper pulp may be incorporated with viscose solution and the mixture applied to a fabric base in quantity sufficient to fill the fabric and give a surface suitable for painting.

D. J. NORMAN.

Manufacture of cellulose esters of inorganic acids. I. G. FARBENIND. A.-G. (B.P. 279,796, 9.8.27. Ger., 26.10.26).—Cellulose esters of inorganic acids are made by treating alkali-cellulose, in the preparation of which more than 1 mol. of caustic alkali per mol. of $C_6H_{10}O_5$ has been used, with an excess of an inorganic acid chloride. The best yield and the highest degree of esterification are realised when the alkali-cellulose is made by impregnating cotton (100 pts.) for at least 4 hrs. with caustic soda solution of 40% strength at about –10° and pressing the mass to 200–300 pts. By suitably varying the conditions the degree of esterification may be controlled with the production of esters which differ as regards their solubility, conductivity, and mechanical properties. *E.g.*, alkali-cellulose prepared as described above from 100 pts. of cotton and pressed to 300 pts. is introduced in small lots into an ice-cold solution of 160 pts. of phosphorus oxychloride in 900 pts. of benzene. The heat of reaction first raises the temperature of the benzene to the boiling point, after which the reaction proceeds smoothly with the production of a tough magma on the bottom of the vessel. After 12 hrs. the product is separated, washed with alcohol and ether, dried *in vacuo*, and dissolved in water. The colloidal solution thus obtained is purified by dialysis, and the cellulose phosphate precipitated by the addition of methyl alcohol. The yield is 80% on the weight of parent cellulose, and the ester contains 16% PO_4 . By pressing the alkali-cellulose to 400 pts. and using as the esterifying agent 220 pts. of sulphuryl chloride dissolved in 1800 pts. of benzene, cellulose sulphuric acid ester may be prepared as a whitish, non-hygroscopic mass soluble in water and containing 60% SO_4 .

D. J. NORMAN.

Manufacture of esters of the cellulose series. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 281,927, 19.4.27).—Cellulose is pretreated with a small proportion (less than 2%) of sulphuric acid in the presence of a diluent, preferably an aliphatic acid, and is then partially esterified with an aliphatic esterifying agent. Esterification is subsequently completed by adding a further quantity of sulphuric acid. Thus, 100 pts. of sulphite-cellulose (94% of α -cellulose) are stirred for 6 hrs. at 20° with 400 pts. of acetic acid (99–100%) and 1 pt. of sulphuric acid. 350 pts. of acetic anhydride (90%) are then added, while cooling and stirring, and the mixture is kept at 20° for 2–4 hrs. until a uniform mass is obtained. Esterification is completed by adding 4–5 pts. of sulphuric acid (dissolved in glacial acetic acid) during 5–10 min. After 15–30 min. at 20–30°, or longer at a lower temperature, a clear viscous solution of cellulose acetate is obtained. This process avoids harmful degradation of the cellulose during esterification.

D. J. NORMAN.

Preparation of a modified cellulose for use in the preparation of cellulose acetate. Soc. LYONNAISE DE SOIE ARTIFICIELLE, and P. A. A. CHEVALET (B.P. 264,181, 10.1.27. Fr., 11.1.26).—The acetylation of cellulose is facilitated by pretreatment of the cellulose with 80–90% formic acid containing sulphuric acid (d 1.83) or anhydrous zinc chloride in the proportion of less than 20% by wt. of the mixture, at room temperature or lower for about 2 hrs. A suitable bath is obtained from sodium formate and sulphuric acid. The cellulose,

after washing etc., is acetylated, sulphuric acid (less than 1% by wt. of the cellulose) being used as catalyst, at room temperature or lower.

B. FULLMAN.

Treatment of cellulose fibres. COURTAULDS, LTD., H. J. HEGAN, and J. H. TAYLOR (B.P. 283,672, 1.11.26).—Cellulose fibres which have been immunised by the action of tannic acid and stannous chloride show a tendering effect on storage. This may be avoided by treating the immunised fibre with a weak alkaline reagent such as soap solution or dilute sodium carbonate solution.

D. J. NORMAN.

Treatment of artificial silk manufactured by the stretch-spinning process. HÖLKENSEIDE G.M.B.H. (B.P. 279,791, 29.7.27. Ger., 26.10.26).—Improved results are obtained by the process of G.P. 397,857, in which the freshly spun threads in hanks or skeins are placed on rotating rollers and subjected to vibrational stretching, if, prior to this treatment, the silk is dried. The silk may be remoistened before or during vibrational stretching. This preliminary drying sets the thread and renders it more resistant, so that a more intensive vibrational stretching treatment is permissible.

D. J. NORMAN.

Treatment of transparent material [celluloid]. C. E. SWETT, Assr. to U.S. ENVELOPE Co (U.S.P. 1,656,303, 17.1.28. Appl., 14.3.23).—Celluloid is coated in succession with alcoholic solutions of tartaric acid and potassium acetate to decrease the reflection of light from the surface, and thus improve the transparency.

T. S. WHEELER.

Cooking of sulphite pulp. G. H. TOMLINSON (U.S.P. 1,657,048, 24.1.28. Appl., 7.12.25).—Wood chips and heated sulphite liquor are introduced into the digester simultaneously and through the same opening, whereby the chips become soaked with liquor and are washed into position.

D. J. NORMAN.

Preparations for waterproofing wallpapers and the like for rendering the same washable. A. SANDERSON & SONS, LTD., and H. A. SIMS (B.P. 284,435, 10.12.26).—The paper is treated with a dilute solution of nitrocellulose. If a slightly glossy surface is desired a small quantity of gum, e.g., 5% of ester gum on the weight of nitrocellulose, may be added. A suitable solution for a matt finish contains 10 lb. of nitrocellulose, 5½ gals. of acetone, 4½ gals. of amyl acetate, 7½ gals. of industrial spirit, 11 gals. of petrol, and 1½ gals. of solvent naphtha.

D. J. NORMAN.

Cooking of vegetable fibre. S. D. WELLS (B.P. 273,663, 25.4.27. U.S., 29.6.26).—See U.S.P. 1,626,171; B., 1927, 438.

Increasing and improving the resistance of spun goods from artificial silk waste. J. A. GRAND (U.S.P. 1,660,123, 28.2.28. Appl., 2.3.27).—See B.P. 253,547; B., 1927, 184.

Apparatus for defibrating cotton seed, the decorticated hulls of cotton seed, or other fibre-bearing seeds, and for segregating and collecting the detached fibres. E. CARSTENSEN DE SEGUNDO (B.P. 285,305, 27.6.27).

Apparatus [bobbin] for the fluid treatment of artificial silk yarns. J. BRANDWOOD (B.P. 261,778, 20.11.26. U.S., 20.11.25).

Papermaking machines. P. OFFENHEIMER, S. BLOCK, and E. OFFENHEIMER (CELLULOSE-FABR. OKRIFTTEL A./M. P. OFFENHEIMER) (B.P. 285,311 and 266,670, [A, B] 15.11.26. Ger., [B], 27.2.26).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Dyeing. Silks and dyestuffs. R. C. HOUCK (J. Physical Chem., 1928, 32, 161—186).—The adsorption theory of dyeing applied to the dyeing of silk with acid and basic dyes, together with the adsorption of tannin by silk, has been studied. The adsorption of hydrochloric acid by degummed silk shows a continuous variation of the amount of acid in the fibre with the concentration of acid in the dye bath, and, furthermore, the amount of Orange II taken up by silk increases with increasing hydrogen-ion concentration of the acid bath. The addition of sodium sulphate in this case reduced the amount of dye adsorbed. With Crystal Violet E the most dye was taken up in neutral solution and then the amount decreased with increasing concentration of the sulphuric acid added. More dye was taken up in acetic acid solution than in a sulphuric acid solution of equivalent strength, and the addition of sodium sulphate in this case caused increased adsorption of the dye. All these results are in accord with the view that the dyeing of silk by acid and basic dyes is an adsorption phenomenon. The apparently contradictory results of dyeing practice are due to the presence of bast soap or boiled-off liquor in the bath, this being shown to decrease adsorption of the dyes mentioned above. The amount of tannin taken up by silk increases with increasing concentration of tannin in the bath, and adsorption is quickest in hot solution. The addition of sulphuric or acetic acid furthers adsorption from dilute, hot solutions. The tannin adsorbed at the ordinary temperature, but not that adsorbed at about 95°, is easily removed by washing. Experiments on the precipitation of tannins by the mineral acids are described and the work of Thomas and Foster (B., 1922, 302) is criticised.

L. S. THEOBALD.

Wetting-out agents and other auxiliary products used in dyeing and calico printing. E. J. RATH (J. Soc. Dyers and Col., 1928, 44, 41—44).—Nekals and Leonils are superior to the Turkey-red oil types of wetting agent because, unlike the latter, they are neither absorbed by the fibre nor affected by the acids, alkalis, and electrolytes used in dyeing processes. Nekals have a high wetting-out power even for greasy materials, and a high emulsifying and solvent action on fat, cotton wax, resin, and natural oils, whilst, by their use, preliminary boiling can be shortened or dispensed with, especially in the case of goods to be dyed, in which a pure white is not essential. Nekal cannot be used with basic dyes since the latter are precipitated by it, but good results are obtained by its use with direct, sulphur, and indanthrene dyes, and indigo. It is also useful in the preparation of sizes and finishes, since the starch solutions are thereby made thinner without loss of adhesive power, wet the yarns evenly, with good penetration, and the size does not dust off. In de-sizing 1—2 g. of dry Nekal BX per litre in conjunction with Biolase reduces the time required by one half. Nekal AEM is of no importance as a wetting

agent, but is an excellent emulsifying agent, saponifiable and unsaponifiable fats, oils, waxes, and resins being emulsified by it without the use of soap. Ramasit I and WD are emulsions of paraffin wax, the former being used for sizing and finishing, whilst the latter, which is more concentrated, is used for waterproofing. Laventine BL is a water-soluble fat solvent, and may be used with any soap for cleaning greasy and dirty materials. The Leonils (similar in chemical constitution to the Nekals) are used in the scouring and dyeing of wool. The Prestabit oils possess excellent fastness to acid, lime, and magnesium sulphate, but (with the exception of the KN preparation) lose their wetting-out property in alkaline solution. Prestabit oil BM is of advantage in grey mercerisation since it imparts excellent wetting-out properties to the sodium hydroxide solutions.

B. P. RIDGE.

Humidity and the fading of dyestuffs on wool. J. J. HEDGES (J. Soc. Dyers and Col., 1928, 44, 52—54).—Patterns of dyed fabric were faded by means of a mercury arc at known humidities between 25% and 100% and compared with corresponding unexposed samples in a Lovibond tintometer. Percentage fading of the predominant colour was then compared with both % R.H. and % regain (the latter being obtained from a humidity-regain curve at the temperature of the experiments). A much simpler relation exists between fading and regain than between fading and humidity, straight-line graphs being obtained in the case of all the colours examined, and is expressed by $F = aR + b$ where F is the % loss of colour, R the % regain, and a and b are constants; the mechanism of the action of the water is not understood.

B. P. RIDGE.

Dyeing with "mangue" extract. SCHIRM.—See XV.

PATENTS.

Dyeing of fabrics. R. R. ROSS (U.S.P. 1,655,973, 10.1.28. Appl., 15.6.27).—Material to be dyed in a variegated pattern is immersed in water, and the excess of liquid is wrung out. The material while still twisted is dipped at different points in a number of dyes and is finally immersed completely in a dye which blends all the colours.

T. S. WHEELER.

Stripping of dyestuffs or colours from dyed or coloured [cellulose acetate silk] materials. BRIT. CELANESE, LTD. (B.P. 260,289, 22.10.26. U.S., 24.10.25).—Cellulose materials, particularly those consisting of cellulose ester or ether, and which are dyed by means of S.R.A. or other water-insoluble dyes, are stripped by extraction with a liquid containing a substance which exerts a swelling action on the cellulosic material and also a substance which is capable of dissolving the dyes which are to be stripped. The temperature and duration of the extraction are dependent on the swelling action of the substance used. Satisfactory swelling agents and solvents include aromatic hydrocarbons, *e.g.*, xylene, benzene, and ethylbenzene, acetone, chloro-derivatives of saturated and unsaturated hydrocarbons such as tetrachloroethane, dichloroethylene, etc., and alcohols such as ethyl and methyl alcohols; aqueous solutions of ammonium thiocyanate also serve as swelling agents. When alcohols are used as swelling agents it is pre-

ferable to restrain their action by the addition of benzene.

A. J. HALL.

Colour printing on [cellulose acetate silk] fabrics having a foundation of silk or wool. ÉTABL. PETIT-DIDIER (ANC. MAISON JOLLY-BELIN) (B.P. 270,657, 25.3.27. Fr., 7.5.26. Addn. to B.P. 256,238; B., 1928, 50).—After colouring the cellulose acetate silk by printing, the whites are cleared by passing the fabric through a bath containing 20—25 g. of potassium permanganate per litre, and then through a solution of sodium bisulphite (*d* 1.116); still more effective clearing is thus attained than that described in B.P. 258,562 (B., 1928, 50).

A. J. HALL.

Weighting of natural silk. R. CLAVEL (B.P. 283,019, 24.3.27. Addn. to B.P. 266,640; B., 1927, 295).—By pretreating the silk with an aqueous solution of an acid or a salt, optionally in conjunction with a protective colloid, weighting baths containing considerably smaller quantities of added acid, or even none at all, may be satisfactorily used. *E.g.*, the material may be first impregnated with a 1% solution of phosphoric acid containing 12 g. of glue per litre and is then treated as in the prior patent. The pretreatment with acid imparts to the silk an increased and more uniform receptivity for the weighting agents.

D. J. NORMAN.

Treatment of fabrics. HEBERLEIN & CO. A.-G. (B.P. 276,352, 19.8.27. Ger., 19.8.26).—In the production of wool-like effects on vegetable fabrics by the action of mercerising agents under conditions which permit shrinkage, improved results are obtained if the fabric is composed of raw fibres, *i.e.*, fibres which have not been freed from their natural incrustants. Mild purification by, *e.g.*, dilute acid or diastase is permissible, but treatment with boiling alkaline solutions must be avoided. The fabric may be bleached before or after mercerisation if the bleaching is conducted at a temperature not substantially exceeding 40° and under carefully controlled conditions.

D. J. NORMAN.

Ornamentation of fabrics made of or containing cellulose esters or ethers. R. CLAVEL (B.P. 269,128, 11.10.26. U.S., 6.4.26).—Metallic and other ornamental effects are obtained on fabrics composed of or containing cellulose esters or ethers by coating or printing the fabric with a paste containing a thickener, a finely-divided metal, pigment, or like powder, and a solvent or swelling agent for the cellulose ester or ether, and fixing the powder on the fabric by, *e.g.*, rapidly drying the coated material. After drying, the fabric may be dyed, preferably at 80—100°, washed, and stretched, the object of the stretching being to cause fracture of some of the partially or completely dissolved cellulose ester or ether threads to give a metallic or other effect on a white or dyed background. The fabric may, if desired, be finally calendered.

D. J. NORMAN.

[Apparatus for] dyeing, cleaning, and mordanting of tissues, felts, etc., and similar operations. SOC. DES CONDENSEURS DELAS (B.P. 280,489, 24.2.27. Fr., 10.11.26).

Bleaching machines. JACKSON & BROTHER, LTD., and C. TAYLOR (B.P. 285,689, 23.4.27).

Improvement of fibres (B.P. 264,529).—See V.
Colouring of rubber (U.S.P. 1,660,213).—See XIV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Hydrochloric acid absorption, its mechanism and the apparatus necessary. B. WAESER (Chem. Fabr., 1928, 101—102).—Data from literature are given, including relations between gas and solution concentrations, calculations of cooler dimensions, back pressures of absorption towers, and various data for different tower packings. For the preparation of very pure synthetic acid, fused silica only must be used, and stoneware, coke, packing rings, etc. excluded. For joints a mixture of pitch with barium sulphate or clay and asbestos is recommended. C. IRWIN.

Catalytic oxidation of ammonia. VIII. L. ANDRUSSOV (Z. angew. Chem., 1928, 41, 262—263).—Views on the mechanism of the reaction between ammonia and oxygen in presence of platinum as previously published are summarised. Nitroxyl is regarded as the intermediate product. The large production of nitrogen at high temperatures and low velocities is not explicable as due to thermal decomposition of ammonia or reaction between ammonia and nitric oxide. It is produced in an explosion zone in front of the catalyst, which zone disappears with increasing gas velocity. The conditions of reaction with excess of oxygen suggest the presence of a layer of oxygen at the catalyst surface, whereby nitroxyl and nitric oxide are formed in preference to di-imide. In absence of a catalyst nitrogen is the chief product of oxidation of ammonia. C. IRWIN.

Detection of traces of soluble bromides. H. BAINES (J.S.C.I., 1928, 47, 11—13 T).—To about 1 c.c. of the solution to be tested, 5—6 drops of fluorescein solution are added, followed by chlorine water (roughly 0.001N-solution of chlorine or sodium hypochlorite in water) added drop by drop with shaking. In the presence of bromide a pink coloration is formed after addition of a few drops of the chlorine solution; excess of chlorine bleaches the solution. By using a blank test the limit of accuracy is about 2×10^{-6} N for potassium bromide. An alternative single-solution method may be used. The method may be made roughly quantitative by colorimetric comparison. In the absence of bromides the test may be applied for the detection of iodides. Large quantities of reducing substances are inhibitive. D. G. HEWER.

Condensation of liquids as mists. PAILLY.—See I. Cyanogen and its compounds in the blast furnace. HAUFE and VON SCHWARZE. Steels for synthesis of ammonia. VANICK and others.—See X. Assay of basic lead carbonate. GLAZE.—See XIII.

PATENTS.

Preparation of neutral to weakly acid water-soluble silicic acid compounds. J. A. VON WULFING and A. BUSCH (B.P. 284,450, 11.1.27).—Neutral to weakly acid products, readily soluble in warm water, and obtainable in dry form, are produced by the combination of alkali polysilicates or water-glass solution with casein or albumin substances of acid character (e.g., nuclealbumins or albuminates) and sufficient alkali hydroxide to form metasilicate. E.g., 100 pts. of casein suspended

in 100 pts. of water are mixed with a concentrated solution of about 4 pts. of sodium metasilicate prepared from water-glass or commercial metasilicate by the addition of sodium hydroxide. On evaporation *in vacuo*, casein sodium metasilicate is obtained. B. FULLMAN.

Activation of silicic acid gel. I. G. FARBERIND. A.-G. (B.P. 263,483, 26.10.26. Ger., 22.12.25).—Silicic acid is first dried until the gel structure is consolidated and then treated in a revolving furnace at 300—600° to obtain a solid product. H. ROYAL-DAWSON.

Manufacture of [colourless] sodium sulphide. B. LAPORTE, LTD. From M. SCHLAUGK, G.M.B.H. (B.P. 284,958, 21.9.27).—Sodium cyanide is added to a hot solution of crude sodium sulphide (*d* 1.22) and the whole is heated to 85—90°, allowed to settle, filtered if necessary, and allowed to crystallise. H. ROYAL-DAWSON.

Treatment of natural alkali salts of secondary and tertiary origin. A. LAMBERT, ASSR. to COSMIC ARTS, INC. (U.S.P. 1,660,561, 28.2.28. Appl., 12.6.24).—The solution of sodium chloride, carbonate, and sulphate obtained by leaching natural salt deposits is cooled to 33° and the deposited salt separated from the chloride mother-liquor. A. R. POWELL.

Production of colloidal copper sulphide [for use as an insecticide]. A. R. GRAVER, ASSR. to GRASSELLI CHEMICAL Co. (U.S.P. 1,657,430, 24.1.28. Appl., 25.10.26).—Copper sulphide is precipitated in any usual manner in presence of a protective colloid, e.g., the residue from the evaporation of waste lye from the manufacture of sulphite-cellulose. T. S. WHEELER.

Modification of glauconite. A. C. SPENCER, ASSR. to PERMUTIT Co. (U.S.P. 1,657,700, 31.1.28. Appl., 26.2.23. Renewed 11.2.26).—Glauconite is baked and treated with an alkali. H. ROYAL-DAWSON.

Apparatus for treating aluminous materials. E. MOLDENKE, ASSR. to MOLDENKE PROCESS CORP. (U.S.P. 1,658,457, 7.2.28. Appl., 27.4.23).—A paste obtained by mixing the finely-divided material with sulphuric acid is deposited in a layer on a conveyor belt. Means are provided for caking the paste by heat during its travel on the belt, and for flexing the belt to free the cake therefrom. The discharged cake passes through grinding and heating devices (cf. U.S.P. 1,457,787; B., 1923, 718 A). H. HOLMES.

Reduction of solutions containing titanium, iron, and eventually other compounds. TITAN Co. A/S. (B.P. 279,786, 2.7.27. Norw., 28.10.26).—In the reduction of such solutions by means of iron or its alloys, the efficiency of the process depends on adjusting the concentration of the solution so that it contains 90—130 g. of titanium dioxide per litre, and keeping the temperature below 60°. H. ROYAL-DAWSON.

[Production of] coloured sulphur. W. H. KOBBE, ASSR. to TEXAS GULF SULPHUR Co. (U.S.P. 1,655,504, 10.1.28. Appl., 10.10.25).—Molten sulphur is mixed with a sulphur-soluble azo dye, e.g., malachite-green, or with a substance, e.g., *p*-nitrophenol, which reacts with sulphur to give a coloured product soluble in the molten liquid. T. S. WHEELER.

Treatment of sulphur. J. W. SCHWAB, ASSR. to TEXAS GULF SULPHUR Co. (U.S.P. 1,656,504—5, 17.1.28. Appl.,

10.12.26).—The process of U.S.P. 1,646,838 (B., 1927, 907) is modified in that (A) silica gel, or (B) diatomaceous earth is used.
T. S. WHEELER.

Burning or subliming of sulphur. H. F. MERRIAM, Assr. to GEN. CHEMICAL CO. (U.S.P. 1,657,545, 31.1.28. Appl. 6.5.26).—A portion of the molten sulphur is periodically drained off when the impurities present have concentrated to a predetermined value in the burner or sublimier.
H. ROYAL-DAWSON.

Production of carbon disulphide. I. G. FARBENIND. A.-G. (B.P. 282,049, 3.12.27. Ger., 13.12.26. Addn. to B.P. 237,716; B., 1925, 759).—In a modification of the prior patent, sulphur is evaporated and superheated in an apparatus in the heating furnace containing the reaction chamber; or the apparatus may be inside the latter, especially if internal electric heating is used.
B. FULLMAN.

Manufacture of substantially pure hydrogen. C. E. PARSONS, Assr. to METAL RES. CORP. (U.S.P. 1,658,939, 14.2.28. Appl., 2.9.26).—Pyrophoric iron, produced by the action of producer gas on ferrous oxide below 600°, is treated with steam (below 700° to prevent the formation of magnetic iron oxide); the hydrogen is recovered and the ferrous oxide produced is used again in the process.
W. G. CAREY.

Drying of gases containing nitrogen oxides. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 284,839, 1.1.27).—Humid gases resulting from the catalytic combustion of ammonia in air are treated, at between 30° and 100°, with or without pressure, with sulphuric acid of such initial concentration that it becomes diluted (60–65% strength) and capable of absorbing only small amounts of the oxides.
H. ROYAL-DAWSON.

Purification of gases. GEN. ELECTRIC CO., LTD., and C. J. SMITHELLS (B.P. 284,808, 25.11.26).—Gases such as hydrogen or argon are freed from oxidising impurities, particularly oxygen and water, by passing them over metallic chromium at 700–850°, preferably after partial purification by passing them over red-hot copper and phosphorus pentoxide.
W. G. CAREY.

Extraction of carbon dioxide from gaseous mixtures. I. G. FARBENIND. A.-G. (B.P. 284,574, 24.10.27. Addn. to B.P. 271,852; B., 1928, 91).—Gypsum liquors are used, after filtration, in the scrubbing towers, instead of the neutral or acid solutions of the prior process, such liquors being obtained in the manufacture of ammonium sulphate from gypsum, ammonia, and carbon dioxide.
H. ROYAL-DAWSON.

Precipitation of zinc carbonate. N. A. LAURY (B.P. 285,260, 7.3.27).—See U.S.P. 1,640,708; B., 1927, 877.

VIII.—GLASS; CERAMICS.

Relationship between chemical composition and thermal expansion of glasses. S. ENGLISH and W. E. S. TURNER (J. Amer. Ceram. Soc., 1927, 10, 551–560).—Thermal expansion data, obtained by the authors' method (cf. B., 1920, 407 A), on various glasses are reported. For soda-silica and soda-alumina-silica glasses an approximately linear relationship exists between the thermal expansion and chemical composition.

Boric oxide glasses do not follow this linear relationship. Soda and potash exert dominating influences on the thermal expansion of glasses. Thus, the calculation of the constitution of a glass from its batch formula is an unwise procedure when dealing with expansion data. The influence of zinc oxide on expansion is masked by that of the soda in soda-zinc oxide-silica glasses. Considering glasses of the approximate composition $6\text{SiO}_2 \cdot (2-x)\text{Na}_2\text{O} \cdot x\text{RO} \cdot \text{R}_2\text{O}_3$ or RO_2 , the curves connecting thermal expansion with chemical composition for lead oxide and barium oxide glasses are identical and linear, whilst lime, magnesia, and zirconia glasses all show the straight-line relationship. The curve for alumina glasses deviates slightly from the linear. New so-called expansion factors, which give values for the coefficients of expansion in close agreement with those observed, are computed.
A. T. GREEN.

Penetrability of various rays through glass.
VI. **Penetrability of ultra-violet rays through window-glass; influence of iron and manganese.** S. SUGIE (Rep. Imp. Ind. Res. Inst., Osaka, 1927, 8, [8], 1–22).—With soda-lime (but not potash-lime) glass, the effect of ferric iron in lowering the penetrability is greater than that of ferrous iron. The presence of a little chromium with the iron is without effect. The effect of manganese is slight up to, but great above, 1%. The penetrability is not parallel to the depth or mode of coloration by manganese compounds.

CHEMICAL ABSTRACTS.

Solubility of glass constituents. W. MEYER (Chem.-Ztg., 1928, 52, 151).—An ether-alcohol mixture after being kept in a glass vessel for 2 years was found to contain 0.001% of selenium as selenious acid; ethyl acetate, pure ether, methyl alcohol, amyl alcohol, and amyl acetate were also found to have the power of dissolving selenious acid from glass. The selenium is derived from the selenium dioxide or sodium selenate used for decolorising glasses containing iron. Jena glass gave no selenium after a 12 months' test.

W. J. POWELL.

Vitreous enamel slips and their control. W. N. HARRISON (J. Amer. Ceram. Soc., 1927, 10, 970–994).—The problem of reproducing enamel slips of definite consistency under working conditions was studied. Laboratory tests were made on three ground-coatenamels, viz., a high-silica, a high-felspar, and a high-borax enamel. A modification of the Bingham apparatus was used to determine the consistency of the slips, and the effect of various factors on the consistency was studied; these factors were: ageing, varying the concentration of certain solutes in the liquid phase of the slip, adding borax at different stages of grinding, and grinding the frits to different degrees of fineness. In a study of the physico-chemical nature of the slip and its influence on consistency, it was found that both the frit and the clay particles were negatively charged, and that a change in consistency was not necessarily accompanied by a change in p_H value. The work was extended to include factory tests, simplified apparatus, consisting of a feed tube 1.3 cm. in diam. and 90 cm. in length and a capillary 0.16 cm. in diam. and 12.0 cm. in length, being evolved for this purpose. The fundamental factors which govern

the consistency of a slip are: kind and quantity of colloidal matter present; the degree of flocculation of this matter; the percentage of water in the slip; and the size of the particles which have to be suspended by the colloidal structure. The practical measures indicated by a consideration of these factors are discussed.

F. SALT.

Ball and china clays. C. W. PARMELEE and T. N. MCVAY (J. Amer. Ceram. Soc., 1927, 10, 598—628).—Thirteen ball clays and seven china clays used in porcelain bodies were examined. Owing to impurities, determinations of the adsorption of malachite-green by ball clays indicate no characteristics of value. With china clays, in general, the greater the adsorption of the dye, the greater is the plasticity of the raw clay and the lower its porosity after a specific heat treatment in the vitrification range. Whilst the experimental electrolytes have no effect on china clay suspensions, ball clays can be divided into four classes according to their behaviour in suspension towards these substances. American ball clays have a greater amount of water of plasticity than English clays. The lowest value for the modulus of rupture of washed unfired clays is given as 284 lb./in.² and the highest as 819 lb./in.² English clays show the higher value for this property. Whilst showing less shrinkage up to cone 12 than American ball clays, the English varieties give indications of overfiring at this heat-treatment. The true sp. gr. tends to increase with increasing mullite content of the fired clay.

A. T. GREEN.

Nebraska clay. A. J. RYANES (J. Amer. Ceram. Soc., 1928, 11, 46—60).—The effect of calcium carbonate on the physical properties of bricks made from ordinary red brick clay was studied. Various amounts of pure calcium carbonate which passed 150-mesh were added to an apparently poor grade of clay, briquettes were made from the mixture, and both fired and unfired specimens were tested. The addition of lime decreased the drying shrinkage and firing contraction at all temperatures. It lowered the crushing strength of mixtures fired at high temperatures, and increased the porosity up to the vitrification point.

F. SALT.

Progress report on investigation of saggar clays.

IV. Elasticity, transverse strength, and plastic flow at 1000°. R. A. HEINDL (J. Amer. Ceram. Soc., 1927, 10, 995—1004).—The data given in Report III (B., 1928, 52) are supplemented by similar data observed at 1000°, together with an investigation of the elastic and plastic properties of the clays and the apparent relation between the rate of plastic deformation and refractoriness (cone equivalent). The range of refractoriness extended from cone 14 to cone 33, but all the clays showed plastic deformation at 1000° with a load producing an outer fibre stress of 30 lb./in.² (approx.). No relation was found between the average plastic deformations and the loads applied. The majority of the clays had a much lower modulus of elasticity at 1000° than at room temperature, though their transverse breaking strengths were greater. The cone equivalents of the clays gave a much better indication of the elastic recovery and plastic flow properties at 1000° than did the chemical analyses. Clays with a cone equivalent

higher than 30 showed little variation in plastic flow; those having an equivalent of 30 and below had greater plastic flow as a group, and the plastic flow was approximately inversely proportional to the cone equivalent. The maximum elongation of the extreme fibre decreased with increase of temperature up to 750°, but at 1000° the deformation at failure was much greater than at 750° and lower temperatures.

F. SALT.

Thermal characteristics of clays. A. E. MACGEE (J. Amer. Ceram. Soc., 1927, 10, 561—568).—The thermal reactions taking place during the firing of clay wares are discussed on the basis of original experimental data, whilst previous work on the subject is reviewed. It is shown that practically all clays undergo an endothermic reaction associated with the evolution of hygroscopic water and the breakdown of the clay molecule between 100° and 650°, which reaches a maximum at 550° and results in the absorption of from 40 to 130 g.-cal./g. of air-dried material. Most clays undergo an exothermic reaction of undefined significance in the neighbourhood of 950° with the evolution of 5—30 g.-cal./g. of air-dried material. Thus, the specific heats of most clays lie between 0.4 and 0.5 over the interval 20° to 1100°. Using such data, the reputed thermal efficiency of kilns is considerably increased.

A. T. GREEN.

Hydrogen-ion concentration and electrical conductivity of clay slips. II. Laboratory study. A. H. FESSLER and H. M. KRANER (J. Amer. Ceram. Soc., 1927, 10, 592—597; cf. B., 1928, 53).—Attempts are made to define the causes of the variations in the hydrogen-ion concentration, the electrical conductivity, and viscosity of certain clay slips. The influences of temperature, vigour and time of stirring, and the presence of salts on these properties are examined. It is shown that the hydrogen-ion concentration decreases whereas the conductivity increases with rising temperature. With casting slips, vigorous stirring affects the viscosity and conductivity values, but does not influence such determinations on plastic slips. The viscosity and conductivity values are increased by the addition of common salt to a slip, whilst the hydrogen-ion concentration is not affected by this treatment. The influences of sodium silicate, aluminium chloride, and magnesium sulphate are also detailed. It is concluded that viscosity determinations give the best available data for control purposes.

A. T. GREEN.

Effect of potash and soda feldspars in china body. E. SCHRAMM (J. Amer. Ceram. Soc., 1927, 10, 1005—1006; cf. Geller, B., 1928, 53).—The behaviour of different feldspars in vitreous bodies is discussed. A china body was made up with pure potash feldspar (A), pure soda feldspar (C), and a mixture of the two (B). Plates and briquettes were biscuit-fired to cone 11 and glost-fired to cone 6 (half down). Draw trials were taken with the briquettes at intervals of 20° and the total linear shrinkage, absorption, and bulk sp. gr. were determined thereon. The absorption figures show that an increase in the total percentage of alkali (i.e., increase in the potash:soda ratio) produces earlier vitrification; this is most marked in bodies with the high soda content. Bodies containing 13% and 7% of potash (A and B)

are similar in translucency and firing range, since both overfire together. F. SALT.

Physical properties of chemical stoneware bodies. A. E. MACGEE (J. Amer. Ceram. Soc., 1927, 10, 569—579).—Nine commercial chemical stoneware bodies were investigated for crushing, transverse, and impact strength, thermal expansion, resistance to heat shock, and elasticity. In making up the test pieces each body was tempered with 20% of water and fired to cone 8 in commercial kilns on a 9-day firing basis. Thermal data concerning the firing of these bodies is given. The results show that the ordinary semi-vitreous chemical stoneware bodies expand about 0.3% between room temperature and 500°. Great variations in the elasticity and crushing strength values were obtained, the elasticity of a highly vitrified material being distinctly less than that of a porous body. The crushing strength varied from 10,000 to 260,000 lb./in.² It is desirable that stoneware bodies have a high tensile strength and temperature diffusivity together with a low thermal expansion and modulus of elasticity. Such a combination gives increased resistance to heat shock. The mechanism of the impact test together with the interpretation of the results are discussed.

A. T. GREEN.

Use of silicon carbide refractories in boiler furnaces. B. M. JOHNSON and J. A. KING (J. Amer. Ceram. Soc., 1927, 10, 1007—1015).—The chief causes of failure of boiler furnace refractories are slag adhesion, erosion, failure of the structure, and spalling, the importance of each of these factors varying with the type of coal used. Silicon carbide refractories tend to prevent adhesion of clinker. For practical purposes the bonded brick is superior to the recrystallised type. The bricks are installed along the fire line. Silicon carbide bricks are rapidly eroded by coal ash having a high content of iron and consequent low m.p., and also by ash containing medium amounts of iron (8—10%), if it is converted into the molten state by the action of hot flames under pressure. These adverse conditions are met by air-cooling and water-cooling. Air-cooling is carried out either by circulating air behind the walls and then forcing it through the wall into the furnace (Bernitz bricks are used for this purpose), or by circulating it behind the wall and then using it as preheated air for combustion. Silicon carbide is well adapted to air-cooled structures, owing to its high thermal conductivity, great strength under load at high temperatures, and high refractoriness. The use of "water walls," for which silicon carbide has been found satisfactory, is a recent development of boiler furnace design. F. SALT.

Petrographic study of some slags from boiler furnaces. S. J. McDOWELL and H. C. LEE (J. Amer. Ceram. Soc., 1928, 11, 35—41).—Specimens of slag from two furnaces burning different kinds of coal were examined. The coal ash contained 19.3 and 29.2% Fe₂O₃ and 8.0 and 2.8% CaO. In the cool slag, plagioclase feldspar and iron oxide are the dominant mineral phases. The interface between slag and refractory consists largely of mullite, with iron in solid solution, surrounded by glass. The slag promotes the growth of small mullite crystals in the refractory, and large needles

are formed in the zone of contact between slag and fire-clay refractory. With high-alumina refractories, a thicker, deeper-stained interface is formed as the alumina content increases. Mullite predominates on the outer surface, but becomes mixed with corundum grains as the refractory is approached. Large crystals both of corundum and mullite appear in the stained area. This zone of crystallisation may be conducive to spalling. F. SALT.

Silica cements. COLE.—See IX.

PATENTS.

Electrical insulators of glass. "OSA" PARTICIPATIONS INDUSTRIELLES SOC. ANON. (F.P. 618,186 and 618,288, [A] 28.6.26, [B] 1.7.26. Ger., [A, B] 1.8.25).—In addition to silica, the glasses contain potassium oxide and sodium oxide and one or more bi- or ter-valent oxides, and, if necessary, boric oxide. Minimum electrical conductivity is obtained when the potash : soda ratio is within the limits 7 : 1 and 3 : 1. The soda may be replaced by iron oxide. F. SALT.

Lead glasses. "OSA" PARTICIPATIONS INDUSTRIELLES SOC. ANON. (F.P. 618,659, 7.7.26. Ger., 1.8.25).—Calcium oxide (2.5—3%) is added to ordinary glass batches containing silica, soda, potash, and lead, to increase their electrical insulating properties. F. SALT.

Manufacture of coherent, granular, non-metallic substances (e.g., glass, enamel). A. UHLMANN (B.P. 255,497, 19.7.26. Ger., 18.7.25).—Finely-divided substances are subjected to the action of an electric or magnetic field whereby they are caused to cohere without direct passage of electric current through them.

J. S. G. THOMAS.

Glass vessels. F. MEYER (B.P. 284,888, 26.3.27).—Glass containers for medicines etc. have imparted to them a tension along a definite line causing them to divide along that line after slight scratching with emery. The tension is produced either by bringing the hot glass into contact with a colder body from within (the other end being used for filling), or by outside chilling after filling. W. G. CAREY.

Continuous kilns. M. BISCH (B.P. 275,616, 29.7.27. Fr., 6.8.26).—In a continuous kiln for pottery etc. the roof slabs are mounted on rollers and rails, so that they can be slid lengthways to expose a chamber for recharging, and a travelling carriage is provided to transfer a roof section from one long set of chambers to another.

B. M. VENABLES.

Ceramic kilns. A. J. COULHON (F.P. 617,468, 12.6.26. Belg., 28.8.25).—An auxiliary kiln is provided outside an ordinary ceramic kiln, and from it hot gases are passed through conduits controlled by dampers into the kiln chambers. Where several kilns are in operation, the waste heat from one may be used to preheat another. The arrangement enables the ware to be dried within the chambers, and it facilitates cooling.

F. SALT.

Combined drying and burning kiln [for ceramic ware]. H. R. STRAIGHT (U.S.P. 1,657,138, 24.1.28. Appl., 14.4.26).—A conveyer passes through a series of drying kilns and a burning kiln in line therewith. A partial load of the plastic ware is delivered to the conveyer

at the outer end of one drying kiln, and the remainder at a point between two of the drying kilns.

H. HOLMES.

Linings for rotary kilns. G. POLYSTIUS (F.P. 618,089, 24.6.26).—Highly refractory materials, such as corundum, zirconium, etc., are mixed with *ciment fondu* and water; blocks etc. formed from the mixture are allowed to set.

F. SALT.

[Preparation of clay for] pottery. C. D. HYTEN (U.S.P. 1,657,997, 31.1.28. Appl., 24.7.24).—Clay is reduced to the consistency of cream, and then combined and ground with a colouring consisting of a ground colour base and a shrinkage-control mixture; after the excess of moisture has been extracted, the plastic mass is ready for use.

H. ROYAL-DAWSON.

Preparation of refractory bodies. VER. F. CHEM. & MET. PROD. (F.P. 618,339, 7.6.26. Ger., 10.6.25).—Magnesia, calcined dolomite, etc., in paste form, are mixed with concentrated colloidal solutions of titanium oxide or titanium salts; products are formed from the mixture, with or without the application of pressure, dried, and fired. The products are denser than ordinary magnesia bricks.

F. SALT.

Apparatus for annealing glassware. BRIT. HARTFORD-FAIRMONT SYND., LTD., Assees. of A. W. RUSSELL (B.P. 279,835, 20.10.27. U.S., 26.10.26).

Production of sheet glass. N. V. MIJ. TOT BEHEEREN EXPLOIT. VAN OCTROOIEN, Assees. of L. VON REIS (B.P. 280,533, 3.11.27. U.S., 10.11.26).

Cooling of glass plates and sheets. Y. BRANCART (B.P. 282,638, 12.11.27. Belg., 21.12.26).

Annealing furnaces (B.P. 283,725 and 283,767). **Flasks** (U.S.P. 1,659,383).—See I.

IX.—BUILDING MATERIALS.

Progress report on the efflorescence and scumming of mortar materials. H. WILSON (J. Amer. Ceram. Soc., 1928, 11, 1—31).—The subject of soluble salts in clays, clay products, and mortar materials, and of the addition of barium salts as a preventive against scumming, is discussed. A study was made of 21 panels built up of red face bricks all made from the same clay, but with different mortar materials, viz., sand, slaked lime, hydrated lime, and Portland cement. Saturated and dry bricks, sea water and fresh water, and machine-mixed and hand-mixed mortars were all tried, and the effect of adding various percentages of barium carbonate, barium hydroxide, and barium chloride was studied. Both temporary and permanent scums, produced by artificial and natural soaking and drying of the walls over a period of more than a year, were noted. All the mortars could be made to scum if treated with sufficient water for a sufficient length of time. Calcium sulphate from Portland cement produces a bad, early scum; other scum-forming salts are calcium hydroxide from the free lime and hydrolysed calcium aluminates and silicates in Portland cement and from lumps of lime in the lime mortars, and, to a minor degree, sodium chloride from sea water. Calcium sulphate is sufficiently soluble to pass into solution through the pores of the brick and spread uniformly over the surface. Both slaked and

hydrated lime should be free of hard lumps, which cause streaks of calcium hydroxide and carbonate. Heavy rain of short duration is not so injurious as soaking drizzle. Under normal conditions, barium carbonate will prevent calcium sulphate scum from Portland cement; 5% was used successfully in public buildings. It is also suggested that diatomaceous earth might combine with the free lime of the mortar materials to produce insoluble calcium silicate and reduce the temporary scum of calcium hydroxide. Preliminary strength tests on mortar materials showed that barium salts had no deleterious effects.

F. SALT.

Effect of lead oxide on hardening of Portland cement. B. GARRE (Z. anorg. Chem., 1928, 169, 305—308).—Even 0.001% of lead monoxide causes considerable retardation in the setting of Portland cement, but with amounts of oxide up to 0.2% the usual period of rapid hardening sets in some hours after mixing. With cement containing 0.5% or 1.0% of oxide, however, this stage in the setting is not observed, and even after 50 hrs. the cement with 1% of oxide crumbles if placed under water. It seems probable that this effect of the oxide is due to its influencing the colloidal changes which apparently exert a preponderating influence on the hardening of cement, for if the cement is heated at 300° for 1 hr. immediately after being mixed the product is equally hard whether lead oxide is present or not.

R. CUTHILL.

Effect of grain size on the properties of silica cement. S. S. COLE (J. Amer. Ceram. Soc., 1927, 10, 644—650).—The variables causing changes in the properties of silica cements are: chemical composition, clay content, and screen analysis. A "rate of settle" test giving indications of workability is developed. It consists in making a slip of the cement with 50% by wt. of water and placing it in a graduated cylinder. The amounts of settle after 8 hrs. and 24 hrs. are obtained. These data agree with practical observations. Tests, including softening point, linear shrinkage, and rate of settle on commercial and laboratory-prepared cements are reported. It is noted that as the fineness of the mixes increases, the softening point is lowered. There is no apparent relationship between screen analysis and rate of settle, probably due to variations in the clay content of the cement. Slow settling cements, in general, have low softening temperatures.

A. T. GREEN.

Failure to harden of concrete mixtures poor in cement. B. GARRE (Z. anorg. Chem., 1928, 169, 301—304).—Examination of the aggregates used for concrete mixtures which, although made with a good quality cement, had failed to set, revealed the presence of up to 14.1% of acid solubles. As mixtures made with a pure aggregate in the same proportions as those employed for the defective mixtures hardened satisfactorily, it was apparently the acid-solubles which prevented setting, the amounts of these in mixtures containing a large proportion of aggregate actually exceeding the amount of cement.

R. CUTHILL.

Consistency of silicate of soda for curing concrete. R. S. BRIGHTLER (Eng. News-Rec., 1928, 100,

316).—The time at which sodium silicate is added to a freshly made concrete surface affects the spreading power, as dilution occurs to a varying extent with varying moisture in the concrete surface. As a result of several trials it is recommended to use sodium silicate solution of d 1.33–1.35 on the day after laying the concrete, which should be properly protected during the intervening night. This gave a complete seal with the least expenditure of silicate. C. IRWIN.

PATENTS.

Cement-kiln system. D. S. JACOBUS, Assr. to BABCOCK & WILCOX Co. (U.S.P. 1,658,443, 7.2.28. Appl., 25.1.23).—Parallel rotary kilns are connected to a common horizontal flue fitted with draught-producing means at a point beyond the kilns. The connexions between the kilns and the flue are such as to equalise the flow of gases through the kilns irrespective of their differing distances from the source of draught.

H. HOLMES.

Building material. G. KNUDSEN (B.P. 284,576, 23.10.26).—Powdered or granular olivine mineral or magnesium orthosilicate of different origin is mixed with magnesium oxide, talc, and binding material, and is heated in moulds to a temperature (1300–1500°) sufficient for the formation of magnesium orthosilicate from the magnesina and talc, but below the m.p. of the olivine.

W. G. CAREY.

Cementitious composition. J. A. McCORMICK and C. A. CABELL, Assrs. to NAT. LIME ASSOC. (U.S.P. 1,656,984, 24.1.28. Appl., 5.9.24. Renewed 28.5.26).—A quick-setting cement comprises calcium hydroxide, calcium aluminate, aluminium sulphate, and calcium carbonate.

T. S. WHEELER.

Manufacture of cementitious material of cellular structure. G. B. HINTON (U.S.P. 1,657,716, 31.1.28. Appl., 19.9.27).—A pulp consisting of hydraulic cement, water, and a froth-flotation reagent is formed and agitated into a froth.

H. ROYAL-DAWSON.

Manufacture of moulded cement bodies. A. CHATELAIN (U.S.P. 1,657,956, 31.1.28. Appl., 22.5.26).—A mixture of cement, sand, water, and glycerin is formed into shapes upon a glossy surface, after which the surface of the shaped object is treated with a solution of sodium silicate and glycerin before it sets.

H. ROYAL-DAWSON.

Preservation of wood. B. R. V. MALENKOVIĆ (U.S.P. 1,656,863, 17.1.28. Appl., 16.12.25; cf. B., 1925, 208).—An aromatic halogenodinitro-compound, e.g., 4-chloro-1:3-dinitrobenzene, is mixed in alcoholic solution with a non-nitrated primary arylamine (1 mol.), e.g., α -naphthylamine, to yield a double compound of value as a wood preservative. The product in the example given has m.p. about 100°. T. S. WHEELER.

Treatment of walls for heat-insulation purposes etc. [by spraying]. E. H. WENZEL (B.P. 284,849, 15.1.27).

Rotary kilns (B.P. 283,669 and 284,494).—See I. **Washable wallpapers** (B.P. 284,435).—See V. **Compositions from pitch** (B.P. 277,291).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Cyanogen and its compounds in the blast furnace.

W. HAUFFE and H. VON SCHWARZE (Arch. Eisenhüttenw., 1927—8, 1, 453–466; Stahl u. Eisen, 1928, 48, 201–203).—Thermodynamical considerations indicate the improbability of any appreciable quantity of cyanogen or of hydrogen cyanide being formed in the blast furnace from the elements or by the reduction of carbon monoxide in the presence of nitrogen. On the other hand, the alkali in the charge should be reduced to free metal; this would immediately combine with excess carbon to form carbide, which takes up nitrogen with the production of alkali cyanide, the greater part of which is volatilised and collects in the upper zones of the furnace and in the flue dust. Secondary reactions may result in the formation of thiocyanate, cyanate, cyanamide, and Prussian blue in the dust chamber. Titanium cyanonitride forms whenever titanium compounds are reduced with excess of carbon in a nitrogen atmosphere, and its formation is accelerated by the presence of manganese sulphide. The volatilisation of potassium cyanide from the blast furnace results in a heat loss of 693 kg.-cal./kg., hence its formation results in a higher coke consumption. Again, it has a highly corrosive action on the walls of the furnace and especially on the tap holes, and appears to be of little value as a reducing or desulphurising agent. A. R. POWELL.

Application of theoretical chemistry to some of the more important processes in the production of steel. H. SCHENCK (Arch. Eisenhüttenw., 1927—8, 1, 483–497; Stahl u. Eisen, 1928, 48, 199–201).—Equations are deduced from the mass-action law to represent the equilibria in the thermal decomposition of carbon dioxide, the dephosphorising of iron, the oxidation of manganese in the converter, and the deoxidation of iron in the converter after the addition of a reducing agent. These equations show that a low temperature and a very basic slag result in the most efficient removal of phosphorus, that the more basic the slag and the lower its content of ferrous oxide the smaller is the amount of manganese oxidised during the period of blow, and that the efficiency of the deoxidation of iron is greater the smaller the solubility of the reducing agent in the molten iron and, except in the case of carbon, the lower the temperature of reduction. A. R. POWELL.

Influence of manganese and the rate of cooling on the separation of ferrite [in steels]. P. SCHAFMEISTER and R. ZOJA (Arch. Eisenhüttenw., 1927—8, 1, 505–510; Stahl u. Eisen, 1928, 48, 312–313).—The carbon content of eutectoidal manganese steels decreases with rising manganese content; thus, with 1% Mn it is 0.83% C, with 2% Mn about 0.77% C, and with 3.1% Mn about 0.71% C. Planimetric measurements of the ferrite areas in steels containing 1–3% Mn with the correct eutectoidal carbon content show that the percentage of ferrite decreases with an increase in the rate of cooling and with an increase in the percentage of manganese, and that the influence of the rate of cooling on the proportion of ferrite is greater the higher the manganese content. For a constant rate of cooling and

manganese content the relation between the proportion of ferrite and the carbon content is a curved line.

A. R. POWELL.

Control of normality in plain carbon steels. F. G. SEFING (Mich. Eng. Exp. Sta. Bull., 1927, [13], 3—25).—Abnormal plain carbon steels (0.15—1.20% C) may be rendered normal by heating at 1093—1107°, regardless of the chemical composition or original micro-structure of the steel, the cementite passing completely into solution.

CHEMICAL ABSTRACTS.

Cementation of ferrous alloys with molybdenum and tantalum, vanadium and cobalt, boron, titanium and zirconium, and uranium. J. LAISSUS (Rev. Mét., 1927, 24, 377—395, 474—484, 591—600, 764—775; 1928, 25, 50—57; cf. B., 1926, 278, 443, 545, 633).—The thickness of the cemented layer produced by heating iron or steel embedded in powdered ferro-alloys of the above metals or in the powdered metals alone increases with the temperature and the time and decreases with a rise in carbon content of the steel. In the case of tantalum and boron the layer consists of solid solution on the inside and a eutectic of iron and an iron-tantalum compound on the outside; with uranium practically the whole of the cemented layer has a eutectiferous structure in which are embedded large crystals of a compound. The structure of the cemented layer of vanadium and chromium in steels is complicated by the presence of complex carbides which cause variations in its thickness. The hardness of the cemented layers after slow cooling decreases in the order: zirconium, titanium, uranium, vanadium, and that of the same layers after quenching from 875° in water in the order: uranium, zirconium, vanadium, titanium, and boron. Cementation with zirconium renders steel practically immune from scaling at 1000°; with chromium the tendency to scale is also very much reduced, but not nearly to the same extent as with zirconium. Rust-resisting coatings are produced only by cementation with tantalum and chromium, whilst with molybdenum, boron, and vanadium the metal is highly resistant to corrosion by 50% hydrochloric acid. Tungsten and cobalt layers resist the action of sulphuric acid (*d* 1.3) but, with the exception of chromium layers, all are violently attacked by nitric acid (*d* 1.15). Cementation with chromium may be effected by electroplating the steel with chromium and heating the metal at 1200° for 10 hrs. whereby a cemented layer of uniform thickness resistant to rusting and to the action of cold dilute nitric acid is produced.

A. R. POWELL.

Deterioration of steels in the synthesis of ammonia. J. S. VANICK, W. W. DE SVESHNIKOFF, and J. G. THOMPSON (U.S. Bur. Stand., Tech. Paper No. 361, 1927, 22, 199—233).—The effects of mixtures of hydrogen and nitrogen containing the equilibrium quantity of ammonia at 500° and 100 atm. pressure on various classes of steels are found to be decarburisation, fissuring, sharp increase in combined nitrogen especially in chromium steels, and porosity in the affected part with consequent lowering of strength and ductility. High-carbon steels are particularly susceptible to decarburisation, even in the presence of 2% Cr, though a steel containing 2.25% Cr and less than 0.3% C is not rapidly

attacked. Addition of tungsten and nickel to such a steel increases the resistance. High-chromium and nickel-chromium steels of the stainless type are most resistant to attack, but the presence of vanadium serves no useful purpose as far as "anticorrosive" properties are concerned. The effects are ascribed to reaction of molecular or activated hydrogen with iron carbide in the straight steels, and to reversible formation and decomposition of iron nitride. In the chromium steels, the tendency to decomposition of the nitride once formed is much less.

L. M. CLARK.

Determination of carbon in pig iron and steel by combustion in oxygen. J. CROCHINA (Z. anal. Chem., 1927, 72, 435—439).—The determination of carbon in high-carbon steels and pig iron by combustion in a current of oxygen at a high temperature usually gives slightly low results owing to partial dissociation of carbon dioxide to carbon monoxide, which is swept out of the hot zone before it can re-combine with the excess of oxygen. To avoid errors from this cause the gases from the combustion tube should be passed through a narrow silica tube heated at 600°.

A. R. POWELL.

Determination of manganese in iron and steel by the silver nitrate-persulphate method. S. W. LIPIN (Z. anal. Chem., 1927, 72, 401—416).—Comparative tests of the following three modifications of the persulphate method of determining manganese in steel have been made: the method of the American Society for Testing Materials, that of the Verein Deutscher Eisenhüttenleute, and Swoboda's method (B., 1924, 635). The last-named method gives very concordant results with an empirical factor up to a content of 1.6% Mn, but requires the longest time for a determination. The second method gives results which approach most closely to the stoichiometric, whereas the American method is inclined to yield erratic figures. The author recommends that the German method be modified as follows: 0.2 g. of steel is dissolved in 15 c.c. of nitric acid (*d* 1.2); the solution is heated to expel red fumes, treated with 50 c.c. of 0.01N-silver nitrate solution and 10 c.c. of a 10% ammonium persulphate solution, heated to 60° during 5 min., cooled in running water, diluted with 50 c.c. of cold water, and, after addition of 3 c.c. of a 1.2% solution of sodium chloride, titrated immediately with arsenite solution until the liquid changes to a pure yellow-green colour. To standardise the arsenite solution, 5 c.c. of 0.03N-permanganate are reduced with nitric acid and hydrogen peroxide and the solution so obtained is treated exactly in the same way as the analysis of a steel is carried out.

A. R. POWELL.

Effect of quenching and tempering on the mechanical properties of standard silver. A. L. NOBURY (Inst. Metals, March, 1928. Advance copy. 17 pp.).—Standard silver (92.5% Ag, 7.5% Cu) as ordinarily annealed consists of a solid solution of copper in silver, with small amounts of a copper-rich solid solution. On heating for $\frac{1}{2}$ hr. at 770°, the copper particles go into solution, and the resulting homogeneous solid solution can be retained as such by quenching. These quenched alloys are about 30% softer and 20—30% more ductile than when in the ordinary annealed condition. On tempering these quenched specimens at temperatures

between 200° and 400°, the excess copper is precipitated as copper-rich solid solution, and structures resembling troostite and martensite are obtained. The tempering increases the hardness by 300%, and the tensile strength by 50%, whilst the ductility falls by about 50%; the change is accompanied by a decrease in volume. Quenched specimens may be hardened by cold-work, and then further hardened by tempering. The tarnishing of standard silver in sulphide or other solutions is accelerated by the presence of oxygen, but the quenched and tempered alloys are more resistant than annealed specimens. The removal of coring in cast cupro-nickel alloys by annealing is greatly accelerated by deformation before or during annealing.

W. HUME-ROTHERY.

Constitution of alloys of magnesium and zinc.

R. CHADWICK (Inst. Metals, March, 1928. Advance copy. 14 pp.).—The equilibrium diagram of the system magnesium-zinc has been investigated by thermal and microscopical methods. The m.p. of zinc is lowered by the addition of magnesium and falls to a eutectic at 368°, the eutectic composition being 7.8 at.-% Mg. The liquidus then rises to a maximum at 585° corresponding to the compound $MgZn_2$, and falls to a eutectic at 342° and 69.8 at.-% Mg, from which it rises to the m.p. of magnesium. A second compound $MgZn_5$ is formed at 380° from liquid and $MgZn_2$, but this change is shown only on heating curves, and not on cooling curves; this compound was not observed by Grube (A., 1906, ii, 355). About 0.3 at.-% Mg goes into solid solution in zinc. The compound $MgZn_5$ forms solid solutions to the extent of about 0.4 at.-% at 370°, but these break down on cooling. The compound $MgZn_2$ forms solid solutions within the limits 32.3–43.2 at.-% Mg at 350–400°; on cooling it undergoes, at 357°, a transformation, the nature of which is uncertain. Solid magnesium dissolves about 3.2 at.-% Zn at 480°, but on cooling most of the zinc is precipitated as $MgZn_2$. W. HUME-ROTHERY.

Minute shrinkage cavities in cast alloys of heterogeneous structure.

W. A. COWAN (Inst. Metals, March, 1928. Advance copy. 6 pp.).—The factors affecting the formation of shrinkage cavities are discussed. Tin-base white metals (containing approximately 7.5% Sb and 7.5% Cu) are practically free from shrinkage cavities, but these are present if small amounts of lead are added. The main constituent of these alloys is a solid solution of antimony in tin which solidifies at 237°, but the presence of lead introduces small quantities of a eutectic solidifying at 183°, and if, while still liquid, small drops of this eutectic are completely surrounded by solid, shrinkage cavities must be formed owing to the contraction on freezing.

W. HUME-ROTHERY.

Analysis of duralumin and other light alloys.

TOURNAIRE (Ann. Chim. analyt., 1928, [ii], 10, 1–5, 33–37).—Copper is deposited electrolytically from a nitric-sulphuric acid solution of the alloy. For the determination of manganese, the sample is dissolved in a mixture of nitric, sulphuric, and phosphoric acids. Silver sulphate is added and, after oxidation with ammonium persulphate, the resultant permanganate solution is titrated with arsenious acid. The use of hydrofluoric acid in the mixture of solvent acids is deprecated since

it may dissolve manganese from the reaction vessel. Magnesium is weighed as pyrophosphate. The alloy is dissolved in sodium hydroxide solution and the residue is taken up in nitric acid. The nitric acid solution is evaporated to fuming with sulphuric acid. After dilution with water, manganese is precipitated as insoluble oxides by oxidation with ammonium persulphate and, together with silica, is collected on a filter. Citric acid is added to hold up iron, copper, and aluminium, and the magnesium present is precipitated with vigorous agitation as magnesium ammonium phosphate. Occasionally, oxidation of manganese by the method described leaves a trace of permanganate in solution. The use of alcohol to destroy this is not advisable since manganous salts are formed and are precipitated as manganese ammonium phosphate. It is better to determine traces of manganese in the magnesium pyrophosphate by redissolving, oxidising the manganese, and titrating as described above. Silicon is determined by dissolving the alloy in a mixture of hydrochloric, nitric, and sulphuric acids, the residue after evaporation to fuming, dilution, and filtration, being calcined and treated with hydrofluoric-sulphuric acid mixture in the usual way. Metallic silicon which may persist is removed with a mixture of nitric and hydrofluoric acids. Iron is separated from the mixture of oxides insoluble in sodium hydroxide solution by precipitation of the sulphuric acid solution with cupferron, or, alternatively, the acid solution of these oxides is reduced by zinc amalgam and the ferrous iron is titrated with permanganate. For the separation of zinc, the sodium hydroxide solution containing zinc and aluminium is treated with slight excess of sodium sulphide. The precipitate is dissolved in hydrochloric acid, and aluminium is precipitated with ammonia. Zinc sulphide is thrown out from the filtrate with ammonium sulphide and is calcined to oxide. Nickel in the mixture of alkali-insoluble oxides is precipitated as the dimethylglyoxime compound from ammoniacal solution, the other metals present being kept in solution by tartaric acid.

L. M. CLARK.

Electrolytic detinning of scrap copper. W. W. STENDER and A. A. IVANOV (Z. anorg. Chem., 1928, 169, 399–404).—Tinned copper may be rapidly and cheaply detinned by using it as the anode in the electrolysis, with an iron cathode, of a 1.2% sodium chloride solution, the completion of the process being indicated by a sharp rise of the *P.D.* between the electrodes. The current density should not exceed 50 amp./m.², and the solution must be kept neutral by the continuous addition of hydrochloric acid. It is, however, somewhat difficult to recover the tin from the solution. This disadvantage is absent if the electrolyte is a 3% hydrochloric acid solution, and the cathode is copper. In this case, copper is separated at the cathode along with hydrogen, and may be melted up for bronze, whilst the solution is readily worked up for a commercial grade of stannous chloride.

R. CUTHILL.

PATENTS.

Metallurgical furnace. A. M. Gow (U.S.P. 1,659,869, 21.2.28. Appl., 17.6.21).—Low-pressure air and gas are supplied separately to a reverberatory furnace by means of two adjacent wide conduits and

caused to intermix as they emerge from the conduits by suitably located jets of compressed air. A. R. POWELL.

Rotary melting furnaces. W. BUSS (B.P. 274,451, 5.7.27. Ger., 16.7.26).—The construction of a rotary furnace is such that the longitudinal axis of the drum is at an angle to the horizontal axis of rotation. An internal shoulder is provided close to the axis so that the contents of the furnace do not flow into the hollow trunnion at any point of rotation, the charging hole being situated also in the shoulder. C. A. KING.

Annealing furnace. R. H. SMITH (U.S.P. 1,656,924, 24.1.28. Appl., 14.3.25).—The material is delivered into one end of a horizontal rotary tube mounted in the heating chamber, is conveyed along the tube by a helical rib, and discharged through peripheral openings between the end of the rib and the other closed end of the tube. The openings increase in size in passing circumferentially from the end of the rib in a direction opposed to the rotation. H. HOLMES.

Ore separator. T. W. AINLAY (U.S.P. 1,658,874, 14.2.28. Appl., 5.10.25).—A settling basin, having a retaining lip extending above its lower face at its open side, contains (and extends above and below) a rotatable bowl in order to catch material thrown from the bowl. J. S. G. THOMAS.

Production of iron from iron ores. J. W. HORNSEY and H. E. COLEY (B.P. 284,040, 26.7.26).—Iron ores or oxides are treated in a number of closed chambers arranged in series as, e.g., a series of connected rotary furnaces. In the first the ore is preheated, in the second it is reduced by contact with carbonaceous material and hydrocarbons in the stage of "cracking," whilst in the third chamber cooling of the metallic iron is effected, all the stages being conducted in a substantially neutral or reducing atmosphere. [Stat. ref. to B.P. 215,400 and 11,224 of 1914.] C. A. KING.

Manufacture of malleable cast iron. H. A. SCHWARTZ, ASSR. to NAT. MALLEABLE & STEEL CASTINGS CO. (U.S.P. 1,660,398, 28.2.28. Appl., 22.10.26).—Molten cast iron is treated with a rare-earth element which has a high affinity for sulphur and which does not interfere with graphitisation; on cooling, the sulphide of the added element separates first from the mass in the form of spherical inclusions. A. R. POWELL.

Manufacture of articles hardened in their marginal layers by nitrogenation. F. KRUPP A.-G. (B.P. 272,182, 7.5.27. Ger., 2.6.26).—Articles made from steel alloys containing, separately or in combination, 4–8% Si (or Mn, Ni, Cr, Mb, W, V, Ti, or Zr), 0.5–2% Al, and up to 0.7% C are surface-hardened by nitrogenation. C. A. KING.

Zirconium-manganese steel. F. M. BECKET, ASSR. to ELECTRO-METALLURGICAL CO. (U.S.P. 1,660,409, 28.2.28. Appl., 9.7.26).—The steel contains 1–2% Mn, less than 1% C, and a substantial proportion of zirconium to render it strong and hard in the air-cooled condition. A. R. POWELL.

Alloy steels for motor valves. R. JARDINE, ASSR. to RICH TOOL CO. (U.S.P. 1,660,925, 28.2.28. Appl., 3.1.22).—An alloy containing 1–1.2% C, 13–15% Cr, 5–7% W, 0.8–1% Mo, 2–2.5% Si, 0.4–0.6 Mn,

and a maximum of 0.035% P and 0.035% S, the remainder being principally iron, is claimed.

F. G. CROSSE.

Preparation of an alloy steel for die blocks etc. G. N. HERMAN, ASSR. to ALLOY STEEL CORP. (U.S.P. 1,660,790, 28.2.28. Appl., 27.5.26).—A steel alloy containing 0.25–2.25% Cr, 0.20–1.50% Mo, and above 1% Ni is moulded into ingot form, heated to 980–1260° while free from internal strains, forged, and finally annealed. F. G. CROSSE.

Manufacture of a steel alloy. T. A. MOORMANN, ASSR. to KINITE CORP. (U.S.P. 1,659,661, 21.2.28. Appl., 11.11.26).—The alloy contains 13% Cr, 1.1% Mo, 0.7% Co, 0.05–0.20% B, and 1.5% C. F. G. CROSSE.

Wet separation of earth, earthy materials, ores, etc. A. C. HOUDJAK (B.P. 284,826, 16.12.26).—The material mixed with water is passed through a horizontal pipe with a branch pipe merging into it from below, the space between the two pipes being open or provided with a suitable screen. The lighter particles are swept upward along the main pipe and the heavier or coarser particles pass downwards into the branch pipe and may be further cleaned by a repetition of the treatment in a series of such pipes. A. R. POWELL.

Manufacture of shaped masses such as briquettes. S. G. S. DICKER. From MALMBRIKETT AKTIEBOLAGET (B.P. 284,418, 8.11.26).—Mixtures of crushed ore, limestone, carbonaceous materials, etc., bonded with tar and/or molasses, together with less than 0.5% of sodium chloride in the form of an aqueous solution, are made into briquettes, which are dried at 150–300° for 1–3 hrs. Such briquettes withstand a temperature of 900° without breaking up, and are therefore very suitable for blast-furnace smelting. A. R. POWELL.

Agglomeration or briquetting of tin ores. METALLBANK & METALLURGISCHE GES. A.-G. (B.P. 275,618, 29.7.27. Ger., 9.8.26).—Fine tin concentrates are mixed with 7–15% of fine, light, tin oxide, such as is obtained by the oxidation of volatilised tin sulphide, and with 4–6% of slaked lime, and the mixture is damped and briquetted in the usual way; during ageing, the briquettes become extremely hard owing to the formation of calcium stannate. A. R. POWELL.

Apparatus for sintering metal ore. P. ANDERSON (B.P. 284,793, 9.11.26).—An apparatus for sintering finely-divided ore concentrates comprises a circular frame carrying a number of sintering pans supported on journals and capable of being rotated about a centrally arranged vacuum chamber. The pans are provided with air pipes extending upwards from the bottom to the level of the journals and connected with the vacuum chamber by means of detachable pipes operating in stuffing boxes. A. R. POWELL.

Reduction of ores. H. WITTEK (B.P. 274,803, 20.11.26. Ger., 26.7.26).—The evolution of heat and reducing gases during the treatment of calcium carbide and nitrogen is utilised directly for reducing ores, e.g., zinc ores, which are mixed with the carbide in the reaction furnace, or reduced in a separate furnace. Calcium cyanamide produced from the mixed batch is

free from residual carbide, and is obtained in a granular form directly available for agricultural use.

C. A. KING.

Treatment [smelting] of ores for the production of metal. D. CROESE (B.P. 284,459, 20.1.27).—The charge of finely-divided ore and fluxes is fed into the top of a vertical shaft-furnace divided into a number of compartments, all of which are heated with suitably disposed gas burners and provided with central double-conical baffles which co-operate with the downwardly tapering throats joining one chamber with the next lower. The first chamber is not heated, but acts as a preheating chamber, and below the lowest chamber is a crucible in which the metal is allowed to separate from the slag.

A. R. POWELL.

Treatment of copper ores. G. W. PRINCE and J. H. ENGLISH, Assrs. to UNITED VERDE EXTENSION MINING Co. (U.S.P. 1,660,817, 28.2.28. Appl., 13.1.26).—Roasted copper ores are leached with water in a vessel through which is passed, at such a rate that the ore pulp is kept in violent agitation, a current of filtered roaster gases containing sulphur dioxide. The upper part of the leaching vat is provided with an electrolytic unit for precipitating the copper as fast as it is dissolved from the ore.

A. R. POWELL.

Manufacture of finely-divided metals from metallic carbonyls. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 284,087, 4.2.27. Addn. to B.P. 269,677; B., 1927, 527).—Metals other than iron can be obtained from their carbonyls by introducing the latter, either singly or mixed together and with or without the addition of iron carbonyl, into a heated chamber in such a way that the decomposition takes place in the hot free space of the vessel. The carbonyls may be introduced in the form of vapour either undiluted or mixed with inert gases; or solid carbonyls may be either fused under compressed carbon monoxide and atomised in the chamber, or dissolved in a suitable solvent (e.g., benzene or another liquid carbonyl) and the solution injected into the chamber. The decomposition may be effected under ordinary, reduced, or increased pressure; the temperature required depends on the nature of the carbonyl, the associated gas (if any), and the pressure employed.

M. E. NOTTAGE.

Production of refractory metals. E. A. LEDERER, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,659,205, 14.2.28. Appl., 22.11.24).—The sulphide of the metal is heated *in vacuo* to its temperature of decomposition, and the sulphur volatilised is removed.

H. ROYAL-DAWSON.

Porous metal bodies. A. E. WHITE. From GEN. MOTORS RES. CORP. (B.P. 284,532, 15.6.27).—A compressed mixture of finely-divided metals which do not alloy or only partially alloy together with graphite and a second deoxidising agent is heated; e.g., a mixture including copper, lead, graphite, and phosphor-tin is heated so that metallic oxides present are reduced by phosphorus, an alloy of tin and copper is formed, and lead is melted.

C. A. KING.

Refining of lead bullion containing other metals. G. K. WILLIAMS (B.P. 267,104, 17.2.27. Austral., 6.3.26).—In carrying out the Parkes process for the

removal of copper, gold, and silver from lead, it has been found that a much more efficient separation of these metals is effected if the lead is fed into deep and narrow cylindrical kettles containing a bath of molten desilverised lead below a layer of molten zinc, the lead layer being maintained at a temperature of about 330–340° and the zinc layer at 700–850°. The lead to be desilverised is fed together with about 18 lb./ton of zinc directly into the hot zinc layer, and the purified lead overflows continuously through a long spout leading to the bottom of the kettle and maintained at 400°. By keeping the lead in the lower part well stirred, any crusts that form on the sides are loosened and rise into the conjugate zinc layer. Two or more of the kettles may be used in series, the crusts from the later kettles being used instead of zinc in the feed to the earlier kettles. Operating in this manner it is possible to obtain a final lead containing only traces of gold and copper and less than 0.2 oz./ton of silver, together with a zinc "crust" assaying over 25% Ag and only about 10% Pb. The process is continuous and requires much less zinc than the usual Parkes process.

A. R. POWELL.

Production [melting] of alloys of the platinum metals. E. HAAGN, Assr. to W. C. HERAEUS G.M.B.H. (U.S.P. 1,660,159, 21.2.28. Appl., 9.2.26).—The material to be melted is placed on a small block of lime against an electrode parallel to the surface of the block, and the other electrode is applied diagonally to the opposite side of the metal mixture.

A. R. POWELL.

Preparation of metals [sodium alloys] for chemical uses. C. A. KRAUS and C. C. CALLIS, Assrs. to STANDARD DEVELOPMENT Co. (U.S.P. 1,655,908, 10.1.26. Appl., 5.4.23).—Molten sodium is added to molten lead or tin in an atmosphere of nitrogen, and the resulting alloy is granulated by being dropped down a tower in which it is cooled.

T. S. WHEELER.

Copper refining. A. G. DE GOLYER (U.S.P. 1,660,220, 21.2.28. Appl., 16.4.27).—Molten copper is deoxidised with a carbonaceous material to reduce its oxygen content to a point just above that at which carbon monoxide is retained in the metal; complete deoxidation is then effected by addition of a metallic reducing agent, thus avoiding the presence of adsorbed gases in the refined metal.

A. R. POWELL.

Manufacture of metals and chemicals by means of iodine. A. A. OSSA (U.S.P. 1,660,884, 28.2.28. Appl., 4.2.21. Renewed 14.7.27).—Material containing a heavy metal (e.g., copper) soluble in sulphuric acid is leached with that acid and the solution treated with iodine and sulphur dioxide to precipitate the iodide of the metal. The precipitate is reduced with a more electropositive metal, and the solution of soluble iodide thus formed is treated to recover the iodine which is used again in the process (cf. B.P. 215,439; B., 1924, 601).

A. R. POWELL.

Recovery of copper from liquors by precipitation. I. G. FARBENIND. A.-G. (B.P. 276,017, 13.8.27. Ger., 14.8.26).—The precipitation of copper from metallurgical liquors is effected in two or more stages by means of zinc hydroxide. The fresh liquor is treated in the first stage with an amount of precipitant insufficient for the complete precipitation of the copper, so that the

precipitate from this stage is practically free from zinc; in the second stage (or in the final stage when there are more than two) the precipitant is added in excess, and the resulting zinciferous precipitate is used as the precipitant in the first stage of treating a fresh batch of liquor. Alternatively, when the precipitation is effected in more than two stages, each of the resulting precipitates except the first may be used to effect a partial precipitation of copper from cupriferous liquors of an earlier stage. M. E. NOTTAGE.

Protection of molten magnesium. H. H. OSBORNE, Assr. to AMER. MAGNESIUM CORP. (U.S.P. 1,657,693, 31.1.28. Appl., 27.10.22).—Molten magnesium and its alloys are brought into contact with products resulting from the heating of glycerin above the m.p. of the metal. H. ROYAL-DAWSON.

Treatment of aluminium-bearing ores. R. W. HYDE, Assr. to DWIGHT & LLOYD SINTERING CO., INC. (U.S.P. 1,655,608, 10.1.28. Appl., 22.12.26).—An intimate mixture of finely-divided bauxite, hæmatite, and coal is ignited in a current of air. Combustion of the coal proceeds autogenously throughout the mass, and sufficient heat is generated to dehydrate the bauxite, a portion of which combines with the ferric oxide to form ferric aluminate; the product melts and forms a cellular cake convenient for further treatment. T. S. WHEELER.

Aluminium alloys. W. SANDER, Assr. to T. GOLDSCHMIDT A.-G. (U.S.P. 1,656,502, 17.1.28. Appl., 30.11.26. Ger., 21.10.25; cf. B.P. 272,706; B., 1927, 606).—Alloys of aluminium with zinc, copper, magnesium, etc. are quenched from a glowing heat, left at room temperature for 24 hrs., and then heated at 130° for 24 hrs. to increase their tensile strength. T. S. WHEELER.

Aluminium alloys. H. SCHORN, Assee. of R. MÜLLER (B.P. 277,701, 19.9.27. Ger., 18.9.26).—Alloys resistant to the action of sea-water and containing 1–6% Mg, 0.05–1% Ti, with remainder aluminium are claimed. F. G. CROSSE.

Solder for aluminium and its alloys. G. SCHULTZ (U.S.P. 1,661,052, 28.2.28. Appl., 14.6.26. Ger., 15.6.25).—The solder consists of 450 pts. of aluminium, 370 pts. of tin, 180 pts. of zinc, 20 pts. of copper, 15 pts. of silicon, 10 pts. of lithium, and 5 pts. of iron. F. G. CROSSE.

Production of metallised surfaces on non-metallic bodies. A. I. G. WARREN, and PRECIOUS METAL INDUSTRIES, LTD. (B.P. 284,786, 5.11.26).—The surface of the substance to be metallised is treated with a suitable etching agent which opens the surface pores, e.g., dilute acetic acid for mother-of-pearl, or sulphur in carbon disulphide for vulcanised articles and casein. The surface is then given a smooth coating of a silver or gold compound made into a paste with water, chloroform, benzene, or other volatile liquid, and finally heated in steam under a pressure of 3 atm. so that the metallising substance becomes thoroughly incorporated with the surface of the article. Reduction to metal may be effected by heating in a suitable reducing atmosphere, or by electrolytic processes, and, if necessary, a smooth

finish may be imparted by polishing and plating with the same or another metal. A. R. POWELL.

Coating metal [with lead alloy]. E. R. MILLRING, Assr. to AMER. MACHINE & FOUNDRY CO. (U.S.P. 1,660,847, 28.2.28. Appl., 19.10.26).—A lead alloy for coating metal is obtained by adding phosphor-lead to molten lead containing a proportion of tin which serves to increase the solubility of the phosphorus in the alloy. A. R. POWELL.

Removal of incrustation formed on metallic surfaces which are in contact with hot milk. R. SELIGMAN (B.P. 284,778, 4.11.26).—Incrustations, regarded as calcium caseinogenate, which form on metal surfaces used for heating milk, are removed by treatment with a weak (2%) solution of orthophosphoric acid containing casein or gelatin as a protective agent against undue corrosion of the metal. C. A. KING.

Alloys and their application to telegraphic and telephonic conductors. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 284,789, 6.11. and 17.12.26).—Alloys possessing high electrical resistance and magnetic permeability in fields from 0.001–2 gauss contain 32–42% Ni, 1–4% Si (or Al), 1–4% Cr (W, Mo, V), the remainder being iron preferably within the limits 55–65%. Up to 1% Mn may be present to improve the working properties. Particular compositions of iron alloys are Ni 36%, Cr 3%, Al 3%, and Ni 36%, Cr 2%, Si 1.3%. C. A. KING.

Electrodeposition of chromium. W. S. EATON (B.P. 284,900, 2.5.27).—An electrolytic bath for the deposition of chromium comprises a 10% solution of chromic acid to every gallon of which is added 1 oz. of a mixture of 94% of sodium hydroxide, 2% of sodium sulphate, 2% of sodium carbonate, and 2% of sodium chloride. The article to be plated with chromium is first cleaned by being made the cathode in an alkaline bath containing an iron or carbon anode. The plating bath is maintained at 43–54°. A. R. POWELL.

Copper-tin-nickel alloy. R. OZLBERGER (U.S.P. 1,661,083, 28.2.28. Appl., 24.3.27. Austr., 3.4.26).—See B.P. 268,798; B., 1927, 560.

Annealing furnaces (B.P. 283,725 and 283,767). **Rotary kilns** (B.P. 284,494).—See I. **Metal containers for alcohol** (B.P. 269,135).—See III.

XI.—ELECTROTECHNICS.

Electrochemical production of graphitic oxide. B. K. BROWN and O. W. STOREY (Amer. Electrochem. Soc., April, 1928. Advance copy. 12 pp.).—Graphitic oxide may be obtained by the anodic oxidation of graphitised carbon electrodes in a neutral or acid electrolyte with an oxidising anion. Dilute nitric acid, with or without the addition of an oxidising salt such as potassium chlorate, is preferred. Impregnation of the electrode with paraffin wax prevents undue disintegration. By using a current density not greater than 10 amp./ft.², and keeping the temperature at 22–25°, a product containing 9–11% of available oxygen is obtained at a current efficiency of about 20%. Graphite scrap can be used as raw material if it is powdered, mixed with molten paraffin wax, and moulded into

electrodes under high pressure (350 kg./cm.²), but since such electrodes tend to swell up and disintegrate with slight rise in temperature, rosin is preferred as a binder; it also has the advantage of being dissolved or destroyed during the electrolysis, so that the resulting graphitic oxide can be used in a dry cell mix without previous washing with an organic solvent. The available oxygen in graphitic oxide can be determined by means of ferrous sulphate; the results are somewhat lower than by the combustion method.

H. J. T. ELLINGHAM.

Graphitic oxide as depolariser in the Leclanché cell. B. K. BROWN (Amer. Electrochem. Soc., April, 1928. Advance copy. 6 pp.).—Graphitic oxide may be used as a depolariser to supplant or supplement the manganese dioxide used in dry cells. Graphitic oxide itself being practically a non-conductor of electricity requires mixing with graphite, but the oxide prepared electrochemically consists of particles with a core of unchanged graphite, and may only require milling in order to make it suitable for use. The preparation of an effective depolarising mix is described. Cells made with a graphitic oxide depolariser have a notably lower, open circuit *E.M.F.* than manganese dioxide cells, and the voltage during discharge is also somewhat lower. But the available oxygen of the graphitic oxide appears to be more completely utilised, and gassing and evolution of ammonia are greatly reduced. The "shelf life" is comparable with manganese dioxide cells, and the weight is less for given volume of depolariser. During discharge the graphitic oxide is reduced to graphite or other form of carbon, so that the conductivity of the mass increases: hence the cells maintain a high short-circuit amperage during discharge. Such cells may be advantageous for certain kinds of service.

H. J. T. ELLINGHAM.

Graphitic oxide. STOREY.—See II. **Conductivity of clay slips.** FESSLER and KRANER.—See VIII. **—Detinning of scrap copper.** STENDER and IVANOV.—See X.

PATENTS.

[Magnetic] separation of material. F. B. MACLAREN, Assr. to C. H. BRASELTON (U.S.P. 1,657,405, 24.1.28. Appl., 15.9.22).—A mixture of metallic and non-metallic materials to be separated is allowed to fall through a long vertical gap between groups of fixed poles of electromagnets energised by alternating current. The magnetic field is such that the metallic constituents are diverted toward one group of poles more than the non-metallic constituents.

H. HOLMES.

Electrical method and apparatus for identifying chemical elements and compounds and biological organisms. M. E. SIMON and J. A. O'CONNOR (B.P. 283,650, 15.10.26).—Substances having different characteristics are distinguished by being brought successively into active relationship with an electrical control circuit, which is caused to operate an electrical response circuit, e.g., an electron emission relay circuit.

J. S. G. THOMAS.

Electrolytic apparatus. H. J. YEAGER (U.S.P. 1,658,872, 14.2.28. Appl., 16.1.25).—An electrolytic cell comprises a container with electrolyte and electrodes,

an electromagnet having a substantially U-shaped core comprising two pole-pieces, and means for producing a magnetic field between them so that motion of the electrolyte is produced.

J. S. G. THOMAS.

Charging of fluids and other substances with ultra-violet rays. E. L. CUNNINGHAM and H. F. MACBETH (U.S.P. 1,659,085, 14.2.28. Appl., 17.1.25).—A fluorescent substance is dissolved in a liquid which is then vaporised and subjected to the action of the rays.

II. ROYAL-DAWSON.

Galvanic cell. A. HEIL (B.P. 262,814, 10.12.26. Ger., 10.12.25).—A narrow, vertical, slidable zinc electrode, immersed only in the upper part of the electrolyte, extends at right angles on either side of a series of parallel positive electrode plates. A chromium alloy is used for the anode and the non-rusting V2A steel for all connexions.

J. S. G. THOMAS.

Dry cell. W. F. HENDRY and V. YNGVE, Assrs. to NAT. CARBON Co., INC. (U.S.P. 1,656,066, 10.1.28. Appl., 23.1.24).—Impalpable graphite for use as a depolariser in dry cells is treated with 10% hydrochloric acid and then with 4% sodium hydroxide solution to yield a product with a reduced ash content.

T. S. WHEELER.

Selenium cell. J. NEALE (B.P. 284,942, 6.8.27).—A non-conducting, heat-resisting, preferably transparent material, e.g., acid glass, is impressed with a number of lines of a solution known as silver-G and containing gold and/or platinum, the lines preferably forming interdigitated grids or combs. After firing the plate at about 525° the process may be repeated a number of times; the plate is finally heated to about 200°, and the interstices between the grids or combs are thereby coated with selenium, and the whole is annealed.

J. S. G. THOMAS.

Preparation of an electron-emitting material. T. P. THOMAS, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,659,175, 14.2.28. Appl., 20.6.22).—A thoriated filament to which phosphorus has been applied is heated, and the phosphorus maintained in intimate contact with the filament.

J. S. G. THOMAS.

Glow-discharge valve. F. SCHRÖTER, Assr. to AMER. TELEGRAPH & TELEPHONE Co. (U.S.P. 1,656,957, 24.1.28. Appl., 30.3.21. Ger., 23.1.18).—The presence of hydrogen (1–3%) in the argon filling of a cold cathode discharge tube decreases considerably the potential necessary to initiate discharge.

T. S. WHEELER.

Electric vacuum valve tube. F. SCHRÖTER, Assr. to AMER. TELEPHONE & TELEGRAPH Co. (U.S.P. 1,656,956, 24.1.28. Appl., 30.3.21. Ger., 29.1.24; cf. B., 1920, 183 A).—A device for protecting circuits against excess voltage comprises a discharge tube containing a small quantity of an inert gas, e.g., argon, and electrodes of an alkali or alkaline-earth metal.

T. S. WHEELER.

Carbon electrodes (B.P. 284,818).—See II. **Glass insulators** (F.P. 618,186 and 618,288). **Coherent granular substances** (B.P. 255,497).—See VIII. **Magnetic alloys** (B.P. 284,789). **Electrodeposition of chromium** (B.P. 284,900).—See X.

XII.—FATS; OILS; WAXES.

Determination of neutral fat in soaps. L. F. HOYT (Oil Fat Ind., 1927, 4, 357—359).—The sample (10—15 g.) is dissolved in hot 94% alcohol (150—200 c.c.) and the solution filtered. Alcoholic potassium hydroxide (0.5*N*) is added to the neutral solution, and the liquid boiled in a reflux apparatus for 30 min., a blank determination also being performed. The liquid is titrated hot with 0.5*N*-acid. Neutral fat = $[\text{c.c. } 0.5N\text{-alkali} \times 28.05/\text{saponif. value}] \times (100/\text{wt. of sample})$. For soaps of unknown fat composition the neutralisation value (mg. KOH per g.) of the separated fatty acids, multiplied by 0.97, is approx. the saponif. value of the neutral fat from which they were derived. CHEMICAL ABSTRACTS.

Titre of New Zealand mutton tallow. A. M. WRIGHT and I. THOMPSON (J.S.C.I., 1928, 47, 13—14 r).—Summaries of analyses for titre of several thousands of tallow from the North and South Islands of New Zealand show that there is a gradual increase in titre as the stock becomes more mature and older. In general, a peak is reached followed by a slight but definite fall, the reason for which is obscure, but the fall is more or less coincidental with onset of winter. A definite relationship between titre and latitude from which the stock came is observable, particularly seen when the seasonal average over seven years is compared with the mean latitudes. The lower the latitude the lower was the average titre, due probably to climatic variations. There are, however, many anomalies and contradictory points which render any one explanation insufficient.

D. G. HEWER.

Maturing process of polymerised and oxidised linseed oils. P. SLANSKY and L. KÖHLER (Chem. Umschau, 1928, 35, 41—44).—From a critical examination of the variation in viscosity with storing of samples of linseed oil, oxidised or polymerised to varying extents, it is concluded that the highly oxidised or polymerised samples exhibit a maturing phenomenon. An unblown oil retained its original viscosity of 4.2", measured on an Oswald capillary viscosimeter, after storing for three months; a sample blown for 6.5 days at 50° gave values of 1' 16.3" before storing, 2' 18.5" after one month, and 7' 11.2" after three months, whilst a sample blown for 9 days had viscosity of 9' 22.2", and gelatinised completely within three months. Comparable results were obtained for linseed oil containing 1% of cobalt linoleate, and for linseed oil polymerised at 290—295° for periods varying up to 18 hrs. The maturing process was followed with the aid of the ultra-microscope. No corresponding variations in constants such as iodine value and refractive index were observed, and it is concluded that the variation in these characteristics with the degree of oxidation or polymerisation is due to chemical rather than colloidal phenomena.

E. HOLMES.

Determination of organically-combined sulphuric acid in sulphonated oils. K. H. BAUER (Chem. Umschau, 1928, 35, 25—26).—Sulphonic acid groups in sulphonated oils can be accurately determined by heating the oil under reflux for 1 hr. with a known amount of *N*-sulphuric acid and back-titrating with standard alkali the total sulphuric acid present. Allow-

ance has to be made for the amount of acid added and for the original alkalinity of the oil (alkali-refining being customary). This is contrary to the views of Herbig, who advocates decomposing the sulphonate with hydrochloric acid in place of sulphuric acid, but results obtained by the two means are shown to agree. Full details of the above method and of the determination of moisture and acid value are quoted.

S. S. WOOLF.

Test for refined olive oils in virgin olive oils. A. BAUD and COURTOIS (Ann. Chim. analyt., 1928, [ii], 10, 11—14, and J. Pharm. Chim., 1928, [viii], 7, 215—218).—See B., 1928, 210.

Vitamin-A in cod-liver oil extract. MUNSELL and BLACK.—See XX.

PATENTS.

Manufacture of a cleansing compound. W. O. NANCE, ASSR. to DELETE CO., INC. (U.S.P. 1,657,893, 31.1.28. Appl., 9.11.27).—A solution of 7 pts. of caustic alkali in 3 pts. of water is gradually added to a mixture of 60 pts. of pale, clean, mineral oil and 15 pts. of fatty acid, agitated and heated to a temperature exceeding 100°. The oil is emulsified with the progressively saponified fatty acid and 15 pts. of a mixture of suitable solvents are added. The temperature is reduced, but is maintained sufficiently high to permit the completion of the saponification and emulsification. The mass on cooling produces a substantially stable non-aqueous detergent paste readily soluble in dry-cleaning liquids.

S. S. WOOLF.

Extraction of fats etc. SOC. ANON. DES ATELIERS ARMAND & DÉCUNE (F.P. 621,328, 2.1.26).—Fats etc. are extracted by means of trichloroethylene, benzene, etc. in a closed circuit, charging and discharging being mechanical.

S. S. WOOLF.

Preparation of concentrate of cod-liver oil rich in vitamin-A and other active principles. K. KAWAI (B.P. 283,265, 5.10.26).—A concentrate of cod-liver oil rich in clinically effective ingredients is obtained by saponifying the oil to the extent of 50—75% of its saponifiable content by means of caustic soda, milk of lime, etc. Vitamin-A and other unidentified active principles are absorbed into the remaining unsaponified oil as the reaction proceeds.

S. S. WOOLF.

Extraction of castor oil. I. G. FARBERIND. A.-G., Assecs. of J. BEHRENS (B.P. 265,212, 27.1.27. Ger., 27.1.26).—Castor oil is extracted from the seed by means of chlorinated hydrocarbons, e.g., trichloroethylene, carbon tetrachloride, methylene chloride, etc., alone or mixed with other solvents, e.g., mixed with a large proportion of benzene.

B. FULLMAN.

Vulcanised or sulphurised oil compositions. A. DE WAELE (B.P. 284,415, 3.11.26).—Elastic masses of vulcanised oil compositions are obtained by subjecting esters of higher unsaturated fatty acids, in which a hydrogen atom of the fatty acid residue has been substituted by an electronegative radical less electronegative than the hydroxyl group, to the action of vulcanising agents such as sulphur chloride, sulphur thiocyanate, dithiocyanogen, etc., which react energetically with

glycerides of unsaturated hydroxy-fatty acids; the reaction is thus moderated. In particular, there are used esters of oxy- or hydroxy-unsaturated fatty acids in which the hydrogen of the hydroxyl group is substituted by acyl groups, *e.g.*, the chlorides, acetates, formates, and carbonates, mixed with esters of other fatty acids. In practice, there are used acetylated castor oil or oxidised or blown oils. Additions of oils, fats, waxes, fillers, diluents, accelerators, or retarding agents may be made.

B. FULLMAN.

Extraction of oil from oily waste, oil filters, etc. P. AUZY (F.P. 619,627, 9.12.25).—The material to be extracted is raised to a temperature of 100–110° by dry steam in a slowly rotating centrifuge. After $\frac{1}{2}$ hr. the steam supply is discontinued, any condensed water being run off, and the centrifuge is accelerated to 360–400 r.p.m. The extracted oil collects in a suitably disposed sump.

S. S. WOOLF.

Viscosity tester for oils (U.S.P. 1,659,534).—See I. **Fatty matter from wool washing** (B.P. 273,642).—See V. **Edible fat** (B.P. 284,368 and U.S.P. 1,656,474).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Rapid testing apparatus for paint. A. V. BLOM (Chem. Fabr., 1928, 102–103).—Two parallel chains are mounted on pairs of cog-wheels 2 m. apart so as to carry between them 50 test sheets 10 × 20 cm. The chains travel at 20 cm./min. and carry the sheets through three heating chambers and one freezing box. Sprays are fitted between the chambers, one of which can be filled with corrosive gases if desired. The strips are also submitted to the action of ultra-violet light and that from a "Sollux" lamp. A typical time of treatment is 5 days, and it is claimed that with this apparatus natural weathering conditions are closely imitated.

C. IRWIN.

Pigments. I. [Basic carbonate- and basic sulphate-white lead.] H. WOLFF (Farben-Ztg., 1928, 33, 1343–1346).—The chemical similarity of basic carbonate- and basic sulphate-white leads is reflected in parallelism in their properties. Comparisons of oil absorptions, drying times of paints containing these pigments, mechanical properties of films, resistance to weathering, etc. support this contention in general; exceptions are indicated in questions of opacity, retention of gloss, and tendency to support rusting (the greater tendency of the carbonate in this connexion being tentatively attributed to carbon dioxide derived from the pigment rather than to impurities therein).

S. S. WOOLF.

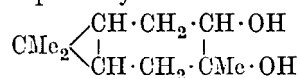
Action of pigments in metal primers. E. J. PROBECK and H. W. BATTLE (Ind. Eng. Chem., 1928, 20, 197).—The ratio of pigment to resin in nitrocellulose primers is of considerable importance from the durability point of view. Whilst a low concentration of pigment—with regard to resin etc.—is held to improve adhesion, 16 months' exposure tests showed that relatively high concentration of pigment produced much greater durability of the whole lacquer system. The compositions of the nitrocellulose primers and surfacers used are discussed.

S. S. WOOLF.

Rapid assay of basic lead carbonate. F. J. W. GLAZE (Chemist-Analyst, 1927, 16, [2], 19–20).—The assay consists of the determination of insoluble impurities, lead as chromate, and carbon dioxide alkalimetrically.

CHEMICAL ABSTRACTS.

Constituents of Indian turpentine from *Pinus longifolia*, Roxb. IV. P. P. PILLAY and J. L. SIMONSEN (J.C.S., 1928, 359–364).—Oxidation of *d*- Δ^3 -carene in acetic acid solution with hydrogen peroxide at 40–60° for 148 hrs. followed by digestion of the non-acidic portion of the product with aqueous alcoholic potassium hydroxide solution, yields *d*-carene- β -glycol, m.p. 90–91°, b.p. 147–150°/18 mm. (+ H₂O, m.p. 75°), together with an isomeric substance, b.p. 155–157°/28 mm., n_D^{20} 0.69° (in chloroform), which, when heated with 5% sulphuric acid for 48 hrs., yields *l*-carene oxide (below) and *p*-cymene. The β -glycol by treatment with phthalic anhydride at 110° yields a *hydrogen phthalate*, m.p. 191–192°, and therefore contains only one secondary alcohol group and probably has the structure



although it is not identical with the glycol of this structure obtained by oxidation of *d*- Δ^3 -carene with potassium permanganate in alkaline solution (Simonsen, B., 1920, 581 A). Oxidation with chromic and acetic acids causes it to undergo complete degradation, no trace of the hydroxyketone being detected. When treated with dilute sulphuric acid, it is converted into *p*-cymene and *l*-carene oxide, b.p. 152°/99 mm., d_{20}^{20} 0.961, n_D^{20} 1.4740, which is not quite pure but contains a small quantity of a ketone isolated as its *semicarbazone*, m.p. 193–193.5°. When the oxide is heated with phthalic anhydride at 160° for 6 hrs. it yields *d*-carene- β -glycol hydrogen phthalate in good yield. When *l*-carene oxide is oxidised with potassium permanganate in acetone at 0°, a complex mixture of acids is produced from which a small quantity of a keto-acid, C₉H₁₃O₃, was isolated as its *semicarbazone*, m.p. 165–166°. Oxidation of *d*- Δ^3 -carene with sodium hypochlorite solution does not proceed smoothly, the products isolated being *hydroxychlorocarene*, b.p. 90–93°/10 mm., d_{20}^{20} 1.0123, n_D^{20} 1.4992, and impure dichlorodihydroxymethylcyclopropylcyclohexane, b.p. 110–113°/10 mm., which is probably a mixture of isomerides.

J. W. BAKER.

Consistency of lacquers. C. D. BOGIN and C. W. SIMMS (Ind. Eng. Chem., 1928, 20, 190–196).—The application of brushing and spraying lacquers is materially affected by consistency. This is evidenced by variation in brushing time or spraying pressure, orange-peel effects, blushing, and general appearance. Factors influencing consistency are concentration of nitrocellulose, composition of solvent mixture, nature of pigment, variation in batches of commercial nitrocellulose, and ageing of lacquer in the presence or absence of impurities. Each of these factors is discussed and data are adduced. Consistency is measured by means of an efflux pipette, glycerol being taken as standard. An empirical relationship is derived connecting consistency with concentration of nitrocellulose.

S. S. WOOLF.

Thinners for nitrocellulose lacquers. J. G. DAVIDSON and E. W. REID (Ind. Eng. Chem., 1928, 20, 199—200).—The requirements of lacquer thinners are:—good solvent properties and freedom from any hydrolysable, noxious, or "blush"-inducing components. "Blush"-resisting solvents require relatively high b.p., but must not unduly retard the setting of the lacquer film. The qualities of amyl acetate, Cellosolve (ethylene glycol monoethyl ether), Cellosolve acetate, and other modern commercial solvents are discussed in relation to their use in thinners. Empirical relationships for deducing the solvent efficiency of various mixtures are described. S. S. WOOLF.

Solvent structure and solvent action. B. K. BROWN (Ind. Eng. Chem., 1928, 20, 183).—The desirability of a solvent for use in cellulose lacquers cannot be computed from its nature alone; thus, it is found that "two-type" solvents (*e.g.*, diacetone alcohol, ethyl ether of ethylene glycol) that will dissolve nitrocellulose and resins separately, will not, without addition of further solvents, give clear lacquers when the solutions are mixed. S. S. WOOLF.

Constants of nitrocellulose solvents. J. A. BRIDGMAN (Ind. Eng. Chem., 1928, 20, 184—187).—The bearing of the following properties of various high-boiling solvents on their use in nitrocellulose lacquers is indicated:—evaporation rate, viscosity of lacquers made therefrom, stability (as measured by rate of hydrolysis in presence of a limited amount of alkali and by development of acidity in presence of small amounts of organic acid), solubility in water, and temperature depression due to evaporation. S. S. WOOLF.

Explosive properties of lacquer-solvent vapours. E. G. RICHARDSON and C. R. SUTTON (Ind. Eng. Chem., 1928, 20, 187—190).—The explosive limits of mixtures of single and mixed solvent and diluent vapours with air, as determined in suitable apparatus (details of which are furnished), are tabulated. It was found that the medium- and high-boiling alcohols and esters are not capable of forming explosive mixtures at ordinary temperatures, but the introduction of even moderate proportions of hydrocarbon diluents renders the air-mixture explosive. In two standard-type spray booths tested, ventilation was found to be adequate to keep vapour concentration much below explosive limits. S. S. WOOLF.

"Crystallising" varnishes and "crackle" varnishes. F. KOLKE (Farben-Ztg., 1928, 33, 1281—1283).—The nature of "crystallising" varnishes and "crackle" varnishes is indicated, the former depending on the drying peculiarities of tung oil and the microscopically wrinkled surface produced under specified conditions, whilst the latter (which are generally nitrocellulose lacquers) owe their characteristic behaviour to low content of non-volatile matter, freedom from plasticiser, and contrast in elasticity with the undercoat. The undercoat is made necessary by the obviously poor adhesion of the "crackle" lacquer, and is preferably not dry when the latter is applied. S. S. WOOLF.

Lacquer formulation, with special reference to the use of cumar. A. ROGERS and C. BANTA (Ind.

Eng. Chem., 1928, 20, 198).—A brief account of the formulation and production of a typical brushing lacquer is given. The advantages of synthetic resins, *e.g.*, cumar, over natural resins in this connexion are stressed, but the use of cumar or ester gum alone as the resin component of a lacquer is not recommended. A combination of the two or of either with damar or other natural resin gives satisfactory results.

S. S. WOOLF.

Camphor oils. VAUBEL and NEDELSCHIEFF.—See XX.

PATENTS.

Production of red lead paints and the like coating compositions. A. FRANKE (B.P. 264,492, 10.1.27. Ger., 15.1.26).—Red lead in which the size of the particles does not exceed 4–5 μ , produced from lead monoxide obtained by a process such as that described in B.P. 189,132 (B., 1924, 343), is used in the manufacture of paints and similar compositions. L. A. COLES.

Marine coating [paint] and the like. G. W. TARR, Assr. to BLUINE Co. (U.S.P. 1,657,438, 24.1.28. Appl., 3.8.27).—A mixture of a metallic pigment, *e.g.*, ferric oxide, a waterproof binder, *e.g.*, tar, and powdered urea is claimed as an anti-fouling paint for ships.

T. S. WHEELER.

Production of white lead. C. H. BRASELTON (U.S.P. 1,655,723, 10.1.28. Appl., 1.4.19).—Powdered lead is treated in a rotating cylinder containing quartz pebbles with carbon dioxide, air, and superheated steam.

T. S. WHEELER.

Production of varnishes etc. C. E. PECZENIK and F. SCHAMBERGER (B.P. 283,233, 8.7.26).—A viscous varnish-body is produced by heating the juice of the *Anacardium* (*e.g.*, the juice expressed from the shells) in the presence of a hardening agent, *e.g.*, borates, peroxides, sulphur-bearing compounds, etc. The thickening action is assisted by the presence of an aldehyde (*e.g.*, formaldehyde) and anti-corrosive substances (*e.g.*, phosphates, chromates). The product is thinned with the usual varnish solvents and the hardening agent is removed, *e.g.*, by filtration. S. S. WOOLF.

Manufacture of primings, varnishes, and paint vehicles. W. T. BRANSCOMBE and R. C. L. EVELEIGH (B.P. 283,998, 24.7.26).—In modification of B.P. 266,401 (B., 1927, 333), a priming material contains as a pore-filler a fatty oil which has been treated with a small quantity of sulphur or sulphur chloride. Thinners and resins, driers, varnish, etc. may be added.

B. FULLMAN.

Manufacture of a colour-binding means. F. SICHEL KOMMANDIT-GES. (B.P. 270,652, 15.3.27. Ger., 10.5.26).—Size-colours are rendered more resistant to rubbing by the addition of oxycellulose or hydrocellulose.

L. A. COLES.

Composition of matter [resin]. M. E. DELANEY and L. T. RICHARDSON, Assrs. to CUTLER-HAMMER MANUF. Co. (U.S.P. 1,655,942, 10.1.28. Appl., 25.7.21).—Naphthalene and sulphur monochloride are heated in presence of a catalyst, *e.g.*, tin or aluminium metal or ferrous sulphide, to yield according to temperature and time of heating a more or less insoluble and infusible resin. T. S. WHEELER.

Production of resinous bodies from formaldehyde and colophony. A. AMANN, Assr. to CHEM. FABR. K. ALBERT G.M.B.H. (U.S.P. 1,658,828, 14.2.28. Appl., 7.8.24).—Colophony is heated with aqueous formaldehyde at a temperature above its m.p. but below that at which foaming occurs. B. FULLMAN.

Preparation of a new type of shellac substitutes. J. SCHIEBER and W. NOACK (U.S.P. 1,660,094, 28.2.28. Appl., 15.4.26. Ger., 29.5.25).—See B.P. 252,715; B., 1927, 148.

Painting grounds (B.P. 284,363).—See V. **Vulcanised oils** (B.P. 284,415).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Coagulation phenomena in *Hevea* [rubber] latex. V. Alcohol, alum, and sodium chloride. VI. Further observations on β -mixture. O. DE VRIES and N. BEUMÉE-NIEUWLAND (Med. Proefstat. Rubber, Buitenzorg, 1927, [23], 497—526, and [24], 527—536; Arch. Rubbercultuur, 1927, 11, [11]).—V. The coagulant effect of alcohol on *Hevea* latex is probably in the main due to dehydrating influence and not to solvent action on the rubber resins or precipitation of the latex proteins. Undiluted latex (1 vol.) is coagulated rapidly by 96% alcohol ($\frac{1}{2}$ vol.), but with latex previously diluted with an equal volume of water more alcohol is required, and with a dilution of 1:9 coagulation with 96% alcohol is impossible. The additional presence of salts, however, facilitates coagulation by alcohol. Sodium chloride itself can coagulate undiluted latex, but with a dilution of 1:9 it is no longer completely effective, although alum is still active even at much greater dilution of latex. With dilute latex the range of concentrations of alum effecting coagulation is narrow, a zone of stability being gained at higher concentrations similar to that already observed with higher proportions of acids; with sodium chloride it is not possible to attain this higher zone of non-coagulation. Alcohol and alum both effect coalescence in latex which has been diluted with water (1:9), heated, and then flocculated by acid; their effect resembles that of thymol or β -naphthol and is distinct in character from that of coalase.

VI. The treatment involved in the production of β -mixture, viz., heating latex diluted with water (1:9) and acidifying, causes a separation of certain serum solids; these, however, are probably without appreciable influence on the coagulation phenomena and do not accelerate vulcanisation. The coagulum obtained from β -mixture by inoculation with fresh latex yields a distinct amount of "coalase" to alkaline water; this possibility favours the view that the coalescence of β -mixture by fresh latex is due to a catalytically active substance or enzyme. The velocity of coalescence is considerably influenced by mechanical factors such as the depth of the layer of flocks.

D. F. TWISS.

Vulcanisation tests of guayule rubber. D. SPENCE and C. E. BOONE (U.S. Bur. Standards, Tech. Paper No. 353, 1927, 22, 8 pp.).—The guayule shrub and its contained rubber are prone to deterioration after the tree is removed from the ground. When steps are

taken to prevent this depreciation the rubber, after being freed from resins by acetone, gives results comparable with those obtainable with plantation *Hevea* rubber and can to a large proportion replace this without appreciable decrease of the tensile product. The permanent set results, however, are high relative to plantation rubber, but the guayule rubber appears to possess a better dispersing action on compounding ingredients. D. F. TWISS.

Influence of "mineral rubber" on the strength of vulcanised rubber. W. ESCH (Kautschuk, 1928, 4, 31—35).—"Mineral rubber" is a mixture of gilsonite with petroleum-distillation residues; it accelerates slightly the vulcanisation of rubber. With careful selection of an organic accelerator and adjustment of its proportion and of the conditions of vulcanisation, the presence of mineral rubber up to 20% (relative to natural rubber) may result in superior tensile qualities. It also facilitates the operations of mixing and extruding or calendering. D. F. TWISS.

Cream from latex. O. DE VRIES and N. BEUMÉE-NIEUWLAND (Med. Proefstat. Rubber, Buitenzorg, 1927, [21], 371—396; Arch. Rubbercultuur, 1927, 11, [8]).—Ammonia-preserved latex when kept for several months gives a cream containing from 70% to 76% of rubber by wt.; after dilution to 35% rubber content this, unlike ordinary latex, resists coagulation by an equal volume of 96% alcohol, but the further addition of a little sodium chloride causes the mixture to coagulate. Acetic acid effects satisfactory coagulation of the cream only after dilution. Ordinary latex, the viscosity of which has been reduced by dilution or by the addition of a small proportion of aqueous ammonia, can be separated, by centrifuging, into two portions containing 3—5% and 55—60% of rubber, respectively. The concentrate from latex containing ammonia may be kept indefinitely. If purified further by re-dispersion of this cream in water and again centrifuging, the rubber obtained is more plastic, vulcanises slowly, and has a tendency to become sticky. D. F. TWISS.

Tackiness in crude rubber. O. DE VRIES and N. BEUMÉE-NIEUWLAND (Med. Proefstat. Rubber, Buitenzorg, 1927, [25], 557—572; Arch. Rubbercultuur, 1927, 11, [12]).—Rubber which develops tackiness after treatment with mineral alkali gradually becomes sticky, weak, and opaque, and collapses to a doughy mass; tackiness caused by hydrochloric acid or by traces of copper is succeeded by the formation of a brittle resinous coating which gradually deepens until the rubber has been converted into a vitreous mass; tackiness induced by sunlight consists in a weakening of the rubber, which, however, does not proceed further if the exposure to light is discontinued. Rubber from ammoniated latex does not exhibit tackiness of the type induced by sodium hydroxide. The changes involved in the development of tackiness are at present obscure. D. F. TWISS.

Aldol- α -naphthylamine [as an anti-oxidant for rubber]. H. KLOPSTOCK (Kautschuk, 1928, 4, 40).—Artificial ageing in a Geer oven indicates that a sample of aldol- α -naphthylamine of German origin is quite as effective an anti-oxidant as "Age-rite" for vulcanised rubber. D. F. TWISS.

Aldol- α -naphthylamine. W. ESCH (Kautschuk, 1928, 4, 40).—The experimental methods in the preceding work are adversely criticised, and doubt is expressed as to the justification of the conclusion drawn.

D. F. TWISS.

Latex contaminated with copper compounds as a source of danger of fire. J. G. FOL and W. DE VISSER (Bull. Rubber Growers' Assoc., 1928, 10, 125—127).—The acceleration of the oxidation of rubber on latex-treated cloth by traces of copper compounds constitutes a distinct fire-risk which, however, can be avoided by ensuring that the ammonia-preserved latex is kept out of contact with any machinery or utensils made of copper alloys.

D. F. TWISS.

Application of the quartz-lamp in rubber laboratories. F. KIRCHHOF (Kautschuk, 1928, 4, 24—27).—The light from a quartz-mercury lamp, passed through a uviol filter so as to include only the ultra-violet rays, causes a characteristic fluorescence in the visible or invisible spectrum with many of the materials used in the rubber industry, and can be used for their identification or grading. Zinc oxide, lithopone, oils, waxes, starch, and natural resins (including rubber resins) can be usefully examined in this way.

D. F. TWISS.

PATENTS.

Production of rubber mixings. K.D.P., LTD., ASSEES. of METALLBANK & METALLURGISCHE GES. A.-G. (B.P. 262,487, 7.12.26. Ger., 7.12.25).—Compounding ingredients, such as barium sulphate, lithopone, silicic acid, calcium oxalate, and barium carbonate, are incorporated in a very fine state of division in rubber by introducing into rubber latex chemically equivalent amounts of soluble materials adapted to form the desired dispersed solid by precipitation. The vulcanised products have high tearing resistance, and it is possible in this way to obtain white or coloured rubber with mechanical properties equal to those of rubber containing carbon black.

D. F. TWISS.

Colouring of aqueous dispersions of rubber. M. C. TEAGUE, ASST. to AMER. RUBBER Co. (U.S.P. 1,660,213, 21.2.28. Appl., 31.3.25).—Aqueous dispersions of rubber are mixed with an emulsion formed by dispersing in water a solution of a water-insoluble dye in a suitable solvent, e.g., sulphonated castor oil.

D. F. TWISS.

Direct production of rubber goods from rubber dispersions. DUNLOP RUBBER Co., P. KLEIN, and A. HEALEY (B.P. 284,736, 4.8.26).—By incorporating in rubber dispersions, e.g., latex, a proportion of protective colloids in excess of that normally employed for preserving purposes, the mechanical condition of the deposits obtained by electrophoresis is improved. Soft soap is suitable for the purpose.

D. F. TWISS.

Manufacture of rubber and rubber articles. DUNLOP RUBBER Co., D. F. TWISS, and F. THOMAS (B.P. 284,829, 20.12.26).—Vulcanised rubber scrap is comminuted, sulphur and an anti-oxidant are then introduced evenly, and the mixture is finally heated under pressure.

D. F. TWISS.

Manufacture of rubber articles. DUNLOP RUBBER Co., and G. W. TROBRIDGE (B.P. 285,113, 14.10.26).—

Rubber articles, produced by electrophoretic deposition on an anodic former, after being dried, are heated, with or without vulcanisation, in a suitably engraved or embossed mould.

D. F. TWISS.

Manufacture of sponge rubber. R. J. NOAR (B.P. 284,938, 19.7.27).—Sponge rubber articles are produced by forming on the surface of the shaped raw mass of mixing a vulcanised skin; this can be effected by the external application of a vulcanising agent active at low temperature. The mass is then heated to cause the internal generation of gas and concurrent vulcanisation throughout the mass. The preformed skin confines the swelling mass and aids the retention of shape.

D. F. TWISS.

Buoyant oil-proof india-rubber. E. YOSHIOKA (B.P. 284,912, 19.5.27).—An oil-resistant india-rubber having d 0.9 (approx.) is produced by mixing raw rubber with factice, sulphur, alkaline soap, and diphenylguanidine, the rubber content of the product generally being at least 80%.

D. F. TWISS.

Vulcanisation of rubber. Preparation of a rubber-vulcanisation accelerator. W. SCOTT, ASST. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,656,834 and 1,656,891, 17.1.28. Appl., [A] 6.5.27, [B] 14.12.25).—A mercapto-compound, e.g., mercaptobenzthiazole, is heated with an organic base (1 mol.), e.g., di-*o*-tolylguanidine, and the product is condensed with an aldehyde (1 mol.), e.g., crotonaldehyde, to yield an accelerator.

T. S. WHEELER.

Artificial petroleum etc. from rubber waste (B.P. 282,565).—See II.

XV.—LEATHER; GLUE.

The "mangue" and its tannin. E. SCHIRM (Bol. Soc. Chim. São Paulo, 1928, 1, 2—7).—The leaves of the "mangue" (*Conocarpus racemosus*, Linn., or *Laguncularia racemosa*, Gärtner), which is a tropical shrub growing in salt marshes on the east coast of S. America and the west coast of Africa, yield a tanning liquid on extraction with hot water. The tannin is probably an ellagotannin. In conjunction with other materials, a hot infusion of the leaves is used for tanning leather, lightening the red colour due to mangrove and quebracho extracts; alone it yields a soft leather of greenish-yellow colour. It may replace sumach in chrome-tanning. By leaching the leaves with hot water or bisulphite solution, and evaporating under reduced pressure, extracts are prepared which are completely soluble in hot or cold water, respectively. The natural liquid extract as manufactured contains 50% of solid matter and 28% of tannin. Owing to the presence of salts the solid extract is hygroscopic. The maximum yield is 5—6% of the weight of fresh leaves in winter. Cotton is dyed in a fast khaki shade by treatment with 20% of the liquid extract, followed by 2—2.5% of potassium dichromate and 2—2.5% of copper sulphate in a second bath. "Mangue" can also be used with direct colours and as a mordant. Wool is dyed directly in a similar shade.

R. K. CALLOW.

Fat-liquoring [of leather]. H. B. MERRILL (Ind. Eng. Chem., 1928, 20, 181—2).—Samples of one-bath chrome-tanned calfskin were fat-liquored with a sul-

phonated neatsfoot oil, dried, split into five layers, and the penetration in each split was determined. The results showed that the oil penetrated more from the grain than from the flesh side, and that the middle split contained natural skin-fat only. No change occurred in the distribution of the fat during the drying out. The amount of oil absorbed increased in direct proportion to the quantity present in the fat-liquor, decreased as the fat-liquor was diluted, and increased with the time up to 4 hrs. and was then constant. Alterations in the p_H value of either the liquor or the leather had little effect on the quantity of oil taken up, but increasing p_H value favoured the penetration of the oil into the skin.

D. WOODROFFE.

PATENTS.

Treatment of hides or skins. H. DODGE (U.S.P. 1,659,520, 14.2.28. Appl., 14.5.27).—Puered or bated skins or hides are agitated with a mixture of alum, salt, and water, then formaldehyde and potassium nitrate are added, and the agitation is continued.

D. WOODROFFE.

Preparation of leather. A. M. KINNEY, Assr. to STANDARD OIL Co. (U.S.P. 1,655,868, 10.1.28. Appl., 23.3.25).—A method of fat-liquoring tanned hides comprises immersing them in an aqueous emulsion of a mineral oil containing the sodium salts of the sulphonic acids obtained by treating mineral oils with sulphuric acid, and a sodium rosin soap.

T. S. WHEELER.

Production of glue from casein. G. LANZENDORFER, Assr. to MARSTIN ADHESIVE Co. (U.S.P. 1,659,271, 14.2.28. Appl., 4.12.24).—Casein is mixed with a liquid containing a phenol, alcohol, water, and at least sufficient alkali to neutralise the acidity of the glue, and heated at 60° until a liquid glue is produced.

B. FULLMAN.

Tanning and dyeing of furs and leather. V. ZETTLITZ and A. PFEIFER (B.P. 281,592, 16.12.26. Ger., 30.11.26).—See U.S.P. 1,649,502; B., 1928, 25.

XVI.—AGRICULTURE.

Influence of reaction on the action of ammonium salts on growth of *Zea mays*. W. MEVIUS (Z. Pflanz. Düng., 1928, 10A, 208—218).—The toxic action of ammonium salts on maize grown in water culture is attributed not to the physiological acidity of these salts, but rather to the harmful influence of the ammonium radicle.

H. J. G. HINES.

Influence of various potash salts on the yield and starch content of potatoes. O. NOLTE and R. LEONHARDT (Mitt. Deut. Landw.-Ges., 1927, 689; Bied. Zentr., 1928, 57, 68—69).—Confirming earlier observations that potassium salts other than sulphates tend to reduce the starch content of potatoes if applied late in the season, "Patent Kali" (mixed sulphates of potassium and magnesium) produced a higher yield of tubers of higher starch content than "40% potash salts" in field trials.

A. G. POLLARD.

Influence of nitrate concentration on the development of barley and the absorption of nitrate from nutrient solution. M. S. MILLER (Mem. Leningrad Agric. Inst., 1926, 3, 57—114).—The absorption of nitrate by barley is largely influenced by the reaction.

Absorption is maximal at the time of heading and blooming, decreasing during ripening and at the end of the vegetation period. The concentration of nitrate indirectly influences the transpiration of moisture.

CHEMICAL ABSTRACTS.

Effect of aluminium sulphate on rhododendrons and other acid-soil plants. F. V. COVILLE (Smithsonian Rep., 1926, 369—382).—Rhododendron, *Franklinia*, and *Vaccinium* plants which were stagnant on rich soil were stimulated to normal growth by treatment with aluminium sulphate solution. Hydrangea flowers were pink when grown on neutral, and blue on treated, soil.

CHEMICAL ABSTRACTS.

Efficacy of mixtures of natural and soluble phosphates, as measured by a bacteriological method, and the effect on the higher plants. G. TRUFFAUT and N. BEZSSONOFF (Compt. rend., 1928, 186, 522—524).—The amount of available phosphate in a mixture of natural and soluble phosphates can be measured by the growth of nitrogen bacteria in a suitable medium containing a known weight of the phosphates. Using this method, verified by experiments on potatoes, maize, etc., it is found that the addition of up to 66% of soluble phosphate to natural phosphate increases its assimilability, but that further addition is useless and even harmful.

B. W. ANDERSON.

Important nitrogen losses during the fermentation and humification of highly nitrogenous plants.

I. Lucerne. J. ZOLCINSKI. **II. Red clover.** J. ZOLCINSKI and A. MUSIEROWICZ (Rocz. Nauk Poln. i Lesnych, 1927, 17, 349—377, 377—396; Bied. Zentr., 1928, 57, 64—67).—I. During the humification of lucerne (in flower) as much as 40% of its total nitrogen is lost in 10 days and 60% in 100 days. Of this loss, approximately one half is as ammonia and the balance as elementary nitrogen. The loss is minimised by leaving undisturbed and avoiding destruction of the covering layer of fungus mycelium. The considerable losses of nitrogen may to a large extent be attributed to physico-chemical processes. It is suggested that plants of moderate nitrogen content build up protein matter differing in constitution from that of legumes, and characterised by the presence of $\cdot\text{NH}_2$ and $\cdot\text{OH}$ groups in the *meta*-position (*i.e.*, the least oxidisable position), and contain also more aliphatic groupings which are quite unoxidisable. There is practically no loss of nitrogen during the humification of plant tissue of moderate nitrogen content.

II. Red clover loses 28% of its total nitrogen during humification, largely as free nitrogen. The formation of nitrite or nitrate during the process was not observed. The carbon:nitrogen ratio of the fermenting mass approaches that of soil humus. The total phosphorus in the humified material does not vary. Temperature and time govern the extent of the humification process. The combination between chalk and humic acids is accelerated by a rise of temperature. Leguminous plants serve as an excellent source of humus substances. Sufficient lime supplies are necessary to complete the humification process, which is very largely a physical and chemical as well as a biological one.

A. G. POLLARD.

Relationship between the sodium chloride content of soils and plant growth. TACKE (Abhandl. des Naturwiss. Vereins, 1927, 26, 503—527; Bied. Zentr., 1928, 57, 56—57).—The sodium chloride content of subsoils is more constant than that of surface soils liable to flooding with saline river water. Variations in plant growth are more closely correlated with the salt content of subsoils than of surface soils. In general, the salt content and natural flora of soils are closely related, and variations in the former bring about periodic changes in the nature of the latter. A. G. POLLARD.

Lime, phosphate, and nitrogen requirements of arable soils. F. ROSSBACH (Forts. Landw., 1927, 2, 450; Bied. Zentr., 1928, 57, 57—59).—Methods for the determination of nutrient requirements of soils are compared. Extracts of soils made with 10% hydrochloric acid indicate the total as distinct from assimilable nutrient contents; but the ratio of (Fe, Al, Ca, Mg): PO_4 is indicative of the relative solubility of the soil phosphate. A 1% citric acid extract of soil gives a measure of the root-soluble nutrients. In the case of phosphates the fertiliser requirement is measured by the ratio of this value to the "total" phosphate value above. The Neubauer process, in spite of careful management, fails to determine the root-soluble phosphate in soil. Satisfactory values for potash are obtained in all but extreme and abnormal types of soils. With further development of the method promising results for the nitrogen requirement of soils are indicated. A. G. POLLARD.

Manurial action of calcium silicate. E. BLANCK, F. GIESECKE, and H. KEESE (J. Landw., 1928, 75, 325—336).—Silicates were without injurious action on growing plants. In fertiliser trials potassium silicate adequately replaced potassium sulphate, and calcium silicate replaced calcium sulphate, lime, or chalk without markedly affecting crop yields. Soluble silica in the form of potassium silicate had no specific effect on plant growth. A. G. POLLARD.

Potash fertiliser action of a mica waste. E. BLANCK, F. GIESECKE, and H. KEESE (J. Landw., 1928, 75, 337—342).—Waste material from the preparation of lithium contained potash as a relatively insoluble silicate. Although exhibiting a definite fertiliser effect, its efficiency was very small compared with soluble potassium silicate. A. G. POLLARD.

Absorption of aluminium and iron by soil. N. D. PRJANISCHNIKOV, JUN., and E. K. LUKOVNIKOV (Z. Pflanz. Düng., 1928, 10A, 232—237).—By determination of the amount of exchangeable calcium replaced from a podsol-loam by solutions of hydrochloric acid, aluminium chloride, and ferric chloride it is found that the adsorption-energy value, e (Gedroitz), for hydrogen ion is not constant but increases with increasing p_H . The values of e for aluminium and iron will form the subject of a further communication. H. J. G. HINES.

Variation of phosphorus content in South African vegetation. J. P. VAN ZIJL (J. S. African Chem. Inst., 1928, 11, 3—11).—The phosphoric acid content of South African grass decreases rapidly with age, and is greater in spring than in summer. A table is given showing the variation in the phosphoric acid

content of several samples of grass of different ages and at the various seasons of the year, as well as the variations in the different parts of the same plant. A. R. POWELL.

The Neubauer method [for determining root-soluble nutrients in soils]. F. MAJEWSKI (Polish Agric. and Forest Ann., 1927, 17, 35; Bied. Zentr., 1928, 57, 60—61).—The Neubauer method does not compare favourably with that of Lemmermann (relative solubility) or with field trials, results obtained being, in general, the same as those arising from direct chemical analysis. Wheat is a more suitable plant than rye for use with the seedling method as it is more sensitive to changes in potash and phosphate concentrations in the soil. A. G. POLLARD.

Determination of nutrient content of a soil by pot experiment. H. WIESSMANN (Z. Pflanz. Düng., 1928, 10A, 206—208).—Pots containing 1500 g. of soil mixed with 5700 g. of glass sand are given a full manuring and are planted with oats. Pots with 7200 g. of sand alone are treated similarly, and by comparison of the two yields a correction is obtained for the physical influence of the soil. By omitting phosphoric acid from the full manuring it is possible to calculate the amount of assimilable phosphate originally present in the soil. H. J. G. HINES.

Determination of soil reaction by means of Merck's universal indicator. H. WIESSMANN and K. STEINFATT (Forts. Landw., 1927, 2, 488; Bied. Zentr., 1928, 57, 49—50).—Values of p_H obtained by the Merck universal indicator agree closely with those determined by Sørensen buffer solutions and by electrometric methods, although the limits of differentiation are much narrower in the latter processes. A. G. POLLARD.

Determination of the phosphate requirement of soils. E. BLANCK (J. Landw., 1928, 75, 343—352).—Comparison is made of methods for determining phosphate requirements of soils involving the "relative solubility" of soil phosphates, the Neubauer seedling method, and field trials. The first-named process yields results most nearly in agreement with plant-culture experiments, the values being more regular, definite, and more quickly obtained than those by the Neubauer method. A. G. POLLARD.

Determination of reaction of peat soils. T. ARND and W. HOFFMANN (Z. Pflanz. Düng., 1928, 10A, 219—232).—A critical examination of the methods in use for the determination of the p_H of mineral soils showed that they could be applied to peat soils with but little modification. A method of procedure to be adopted with the quinhydrone electrode is recommended, which can be used either with water or potassium chloride suspensions. H. J. G. HINES.

Deterioration of strophanthus seed. PICKERING.—See XX.

PATENTS.

Drying of agricultural produce. H. WADE. From A. J. MASON (B.P. 283,014, 9.3.27).—The crop in the form of a mat is passed on a perforated conveyor through a long chamber or drying oven, in which the space on

the upper side of the mat is divided into two compartments. Drying gases heated to about 150° pass downwards from one compartment through the crop mat at a point near the outlet end of the chamber where the mat is in a highly dried condition, thence for a substantial distance along the chamber in a direction opposite to that of the movement of the mat, and finally upwards through the moist portion of the mat at the inlet end of the chamber to the second compartment, whence they escape to the atmosphere. The dried crop mat is then thoroughly disintegrated mechanically.

F. R. ENNOS.

Production of manure from spent molasses waste. E. BAUER and L. SZOLD (EISLER & SZOLD) (B.P. 280,219, 3.11.27. Austr., 5.11.26).—Spent molasses waste is concentrated to $d 1.26$ — 1.32 , cooled below 60°, and intimately mixed with aqueous lime sludge or with carbonated lime. The pasty mass is dried at 100—105° to a moisture content of 7—8%, and broken up to a fine powder which is non-hygroscopic and should contain about 30% of lime.

F. R. ENNOS.

Manufacture of chemical manures. RHEINANIA-KUNHEIM VER. CHEM. FABR. A.-G., and H. BRENEK (B.P. 265,197, 25.1.27. Ger., 1.2.26. Addn. to B.P. 242,512; B., 1926, 71).—The process of heating mineral phosphates, silicates, and alkali carbonates or alkali-containing rocks, *e.g.*, leucite or phonolite, is carried out in the presence of steam, a reducing substance, *e.g.*, coal, being added if the phosphate contains alkaline-earth sulphates. Fuels rich in hydrogen may be used to supply the steam.

W. G. CAREY.

Manufacture of manure. E. W. M. HAMMEL (B.P. 284,741, 27.9.26).—Substances (*e.g.*, bone meal) containing or capable of yielding albuminoids or peptones by the action of an inorganic acid other than sulphuric acid are treated with hydrochloric acid, a potassium fertiliser is added, the whole concentrated to a rubber-like consistency, neutralised with ammonia, and dried at 100—120° *in vacuo*.

H. ROYAL-DAWSON.

Manufacture of fertiliser material. K. R. LINDFORS, ASSR. to MICHIGAN SUGAR CO. (U.S.P. 1,657,128, 24.1.28. Appl., 12.2.26).—Hot dehydrated peat is mixed with hot distillery waste-liquor, and the product is kept for 48 hrs. and then mixed with phosphate rock.

T. S. WHEELER.

Insecticide. R. P. SOULE, ASSR. to COMBUSTION UTILITIES CORP. (U.S.P. 1,655,573, 10.1.28. Appl., 23.6.24).—The use of the heterocyclic nitrogen bases present in low-temperature tar is claimed.

T. S. WHEELER.

Copper sulphide as insecticide (U.S.P. 1,657,430).—See VII. **Reduction of ores** (B.P. 274,803).—See X.

XVII.—SUGARS; STARCHES; GUMS.

Clarification of juice in the cane sugar industry [in white sugar manufacture in Java; presence of glucose in cane molasses]. H. I. WATERMAN (Chim. et Ind., 1927, 18, 758—767).—De Haan's carbonatation process, in which lime and carbon dioxide are added simultaneously to the juice at 55°, shows an economy in lime amounting to 40% compared with the ordinary procedure, besides which filtration is

more easily effected and less sugar is left in the press-cakes. Similarly, in Harloff's hot-sulphitation process the juice at 70—80° is treated simultaneously with lime and sulphur dioxide, after which it is heated to 100° and allowed to subside without filtration. Glucose has been determined in the molasses from different countries by measuring the reducing power of the residue left after fermentation; Javan molasses (sulphitation) gave 6.2%; Cuban (defecation) 9.9%; and Egyptian (sulphitation) 6.1%.

J. P. OGILVIE.

Relation between alkalinity and electrical conductivity of carbonatation juices. V. STANEK and K. SANDERA (Z. Zuckerind. Czechoslov., 1927, 52, 209—213).—Determinations of the electrical conductivity carried out during the operation of carbonatation lead to the conclusion that the former is in direct proportion to the latter, and that the effect of the varying composition of the beet, or of the diffusion water, does not generally exceed the experimental error of 0.01—0.02% CaO. In principle, therefore, it should be possible to use electrical conductivity as a means of measuring the termination of the first carbonatation.

J. P. OGILVIE.

Influence of over-saturation [in the carbonatation process] on the precipitation of some acids in presence of magnesia and alkalis. R. KARGL (Z. Zuckerind. Czechoslov., 1928, 52, 253—259).—Precipitation of the vegetable acids present in beet juices during carbonatation after having added lime containing magnesia depends on several factors, but especially on the alkalinity of the carbonated juice. The presence of alkalis has an adverse effect, lowering the efficiency of the clarification and causing over-saturation. Magnesia exerts no influence so long as its amount does not exceed that of the lime used.

J. P. OGILVIE.

PATENTS.

Preliminary treatment of raw sugar, after-product sugar, etc. before refining. RAFFINERIE TIRLEMONTAISE SOC. ANON. (B.P. 276,611, 2.11.26. Ger., 26.8.26).—The sugars are placed in store rooms the temperature and moisture content of which are so controlled that the syrup surrounding the sugar crystals attains and maintains a low limit of purity, *viz.*, 60 pts. of sugar to 100 pts. of total solids, below which it is not economical to attempt further extraction of sugar from the syrup. The whole is then centrifuged at a force 1600—6000 times the weight of the mass treated, and the separated sugar crystals are passed to the refinery whilst the syrup is used as molasses.

F. R. ENNOS.

Manufacture of grape sugar [dextrose]. W. B. NEWKIRK, ASSR. to INTERNAT. PATENTS DEVELOPMENT CO. (U.S.P. 1,658,998, 14.2.28. Appl., 6.8.24).—A starch-converted dextrose solution is crystallised to flocculate crystalloidal impurities. The crystallised material is melted, and recrystallised with agitation.

B. FULLMAN.

Manufacture of laundry starch. L. O. GILL, ASSR. to A. E. STALEY MANUF. CO. (U.S.P. 1,656,190. 17.1.28. Appl., 25.4.23).—A starch solution which remains fluid at room temperature is obtained by mixing in solution two starches, one of which has been more

vigorously treated with sulphuric acid than the other, but neither of which has been entirely converted.

T. S. WHEELER.

Crystallisation mixer for treating massecuites in sugar works etc. L. F. GARDNER. From SCHNEIDER & Cie. (B.P. 284,954, 8.9.27).

Centrifugal apparatus (B.P. 280,167).—See I. **Manure from molasses waste** (B.P. 280,219).—See XVI.

XVIII.—FERMENTATION INDUSTRIES.

Production of *n*-butyl alcohol, acetone, and methyl alcohol from maize. H. TROPSCH (Brennstoff-Chem., 1928, 9, 1—2).—In two American works a total of 635 tons of inferior maize is daily converted into about 55 tons of *n*-butyl alcohol, 27 tons of acetone, and 9 tons of ethyl alcohol, whilst at one of the works 13 tons of methyl alcohol are simultaneously synthesised from the fermentation gases. The maize, containing about 8% of starch, is mixed with water at 71°, sterilised with steam under pressure, and fermented by means of the micro-organism *Clostridium acetobutylicum*. The fermentation requires about 2½ days, and its progress is followed by determining the acidity of the mixture and the gas evolution. The mixture of alcohols and acetone is concentrated and fractionally distilled. The mixture of carbon dioxide (60%) and hydrogen (40%) liberated during fermentation is washed with water under pressure, to bring the proportion to 25% and 75%, respectively, and is then compressed to 300 atm., and passed over a catalyst comprising 97—75% of zinc (or magnesium) oxide with 3—25% of iron hydroxide. From the resultant mixture of 68% of methyl alcohol and 32% of water pure methyl alcohol is obtained by rectification. W. T. K. BRAUNHOLTZ.

Presence of glycuronic acid in wines made from diseased or rot-infested grapes. D. CHOUCHAK (Compt. rend., 1928, 186, 520—522).—Considerable amounts of glycuronic acid are present in musts and wines from rot- and mould-infested grapes. The acid may be isolated by means of its osazone or by precipitation with ammoniacal lead subacetate. Musts and wines from healthy fruit contain only traces of this acid, and thus the amount detected in a wine gives some indication of the care with which the grapes have been selected. Glycuronic acid in a wine falsifies the determination of dextrose and of several organic acids.

B. W. ANDERSON.

Hemicellulose-splitting enzyme (cytase) of malts. H. LÜERS and W. VOLKAMER (Woch. Brau., 1928, 45, 83—87, 95—99).—The action of aqueous extract of green malt upon xylan prepared from elder pith by the methods of Salkowski and E. Schmidt are studied. The xylan was dissolved in alkali and dialysed till neutral. The temperature and acidity optima are 45° and p_H 5.0, respectively. The rate of action increases with increasing amounts of enzyme, but not in direct proportion, the xylose produced having an inhibiting effect. Under the most favourable conditions about 70—75% of the xylan is hydrolysed in 48 hrs. The enzyme is destroyed by heating in solution for 15 min. at 60°,

but not in presence of xylan. The xylose was identified by its rotatory power and osazone, and was also obtained from xylan prepared from barley. Xylose, but not xylan, was found to be present in finished malt. It was found possible to concentrate the enzyme by adsorption on alumina (optimum p_H 5.0) and removal by phosphate solution (p_H 8.3). Since by twice repeating this procedure the cytase strength of a preparation was increased 21 times, while the diastatic power of the same preparation only increased sixfold, the authors conclude that the two enzymes are distinct.

F. E. DAY.

Extension of malt analysis by a flavouring-test. H. KROFF (Woch. Brau., 1928, 45, 87—88).—If 200 c.c. of the laboratory extract are carefully evaporated to 30 c.c., and a drop of the syrup is applied by a glass rod to the tongue, marked differences of flavour will be noted. The flavour is much affected by malting conditions; e.g., a bitter flavour results from early and slow withering of the rootlets due to excessive aeration. To obtain the best malt, which under the conditions of the test gives a honey-like flavour, the rootlets should be unwithered at the completion of modification, and kilning should be sufficiently rapid to dry the rootlets before their fluids can be reabsorbed by the grain. For this the author considers two 12-hr. periods on a two-floor kiln as suitable.

F. E. DAY.

Yeast as fodder. STICH. **Fermentation of bread.** ELION.—See XIX.

PATENT.

Apparatus for determining the alcohol in a liquid. B. STEIN (U.S.P. 1,658,950, 14.2.28. Appl., 15.4.27).—The apparatus consists of a water-bath (with thermometer), into the air space of which extends a receptacle for the liquid under test, carrying a thermometer and condenser. The percentage of alcohol may be determined when the columns of both thermometers become stationary.

B. FULLMAN.

XIX.—FOODS.

Wheat and flour studies. XII. Factors influencing the viscosity of flour-water suspensions. II. Effect of hydrogen-ion concentration during extraction or digestion period. A. H. JOHNSON and B. L. HERRINGTON (Cereal Chem., 1928, 5, 14—44).—Using the procedure of Gortner, maximum viscosities were obtained by extracting at p_H 4.34—5.07, when the extraction was carried out at 25°. With extraction at 40°, the maximum viscosity was obtained at p_H 4.90—5.44. The proportion of the total nitrogen extracted was about 50% in the case of patent and first-clear flours, and about 35% in the case of second-clear. E.g., a patent flour with distilled water at 25° gave an extract of p_H 5.75, viscosity 155° (MacMichael), and 31.9% of the total nitrogen was extracted. The maximum viscosity (260° MacMichael) was obtained at p_H 4.77, 51.5% of the nitrogen being extracted. The corresponding second-clear flour with distilled water gave p_H 6.20, viscosity 76° (MacMichael), and total nitrogen extracted 37.4%; at p_H 4.39 the values obtained were 188° (MacMichael) and 32.4%. Comparison of flour before and after bleaching with chlorine showed that

the same maximum viscosity was obtained. The increased viscosity observed in water-extracted, bleached flours is not due to oxidation of protein. The decrease of viscosity on extracting at successively lower p_H values frequently showed a marked irregularity at about p_H 8, attributed to the increase of water-imbibing capacity of the protein before dissolution by the hydroxyl ions more than balancing the effect of decreasing extraction of electrolytes. The extraction of electrolytes was separately investigated, using a resistivity method, and it is suggested that an important factor is the liberation of phosphates by phytase. The very much lower viscosities given by flours digested but not extracted at various p_H values may be due in part to this. The results of such experiments were parallel to those with extracted flours. Small quantities of salt depressed the viscosity. From the values observed for the solubility of the proteins, the isoelectric points for those of patent, first-clear, and durum flours at 25° are between p_H 6.2 and 6.5, and for second-clear between p_H 5.5 and 6.0. At 40° the isoelectric points are slightly more to the alkaline side.

F. E. DAY.

Wheat and flour studies. XIII. Relation between the fusibility of flour ash and its mineral constituents. A. H. JOHNSON and S. G. SCOTT (Cereal Chem., 1928, 5, 56—64).—Flour ashes, burned off at 585° for 16 hrs., were classified as fluffy, partially fused, and completely fused. The three classes showed little difference in lime or magnesia content, but the fluffy ash contained about 33% K_2O and 48% P_2O_5 , and the completely fused ash about 26% K_2O and 54.5% P_2O_5 , the partially fused ash being intermediate. Three flours giving the three types of ash, when incinerated for 16 hrs. at various temperatures, gave substantially the same ash contents at all temperatures from 485° to 700°, though at 485° the fusible ash was black and fluffy. At about 700° the normally fluffy ash fused, and at high temperatures all three lost weight slightly. The phosphorus in flour ash incinerated at 585° exists mostly as pyrophosphate; in fluffy ashes a small part is present as orthophosphate, in fused ashes a little metaphosphate is present.

F. E. DAY.

Refractive indices of aqueous and alcoholic extracts of flour. D. W. KENT-JONES and A. J. AMOS (Cereal Chem., 1928, 5, 45—56).—No relation was found between the refractive index of an aqueous flour extract and its soluble extract, gassing power, or ash content, nor between the protein precipitated and the change of refractive index on heating such an extract. The increase of refractive index over that of the solvent is chiefly due to gliadin in 70% ethyl alcohol extracts, and though other substances have a slight influence on the refractive index, this has no relation to the gassing quality of the flour. The refractive index of methyl alcohol extracts appears to give no useful information, and is but slightly affected by the glutenin. The equation to the concentration-refractive index curve for gliadin in 70% alcohol at 20° is $\mu_x = 1.3634 + 0.0018C$, where μ_x is the refractive index of gliadin solution at 20° and C is the concentration of gliadin in g./100 c.c.

F. E. DAY.

Viscosity of flour suspensions. G. VAN DER LEE (Cereal Chem., 1928, 5, 10—13).—A theoretical discussion of the inapplicability of Einstein's equation for the viscosity of colloidal solutions to the viscosity of flour suspensions as measured in the MacMichael viscosimeter.

F. E. DAY.

Aid in the determination of flour strength. F. L. ENGLEADOW (Cereal Chem., 1928, 5, 1—9).—Flat portions of dough, 2.5 in. in diam. and 0.2 in. thick, prepared and rolled under standard conditions from the unknown and a standard flour, are lightly clamped over the open ends of two tubes, of 0.8 in. in diam. The tubes communicate with a vessel from which the air can be exhausted. As this occurs, the discs of dough become distended inwards, and finally the weaker bursts. By repeating the observations it is possible to observe fine differences of strength. Thus, in mixtures of Yeoman and Little Joss flours, 10% variations in the proportions could readily be distinguished. The method, though only comparative and not universally applicable, gave results which compared well with the baking tests, and requires a much smaller sample.

F. E. DAY.

Relation of water-absorbing capacity of flour to protein content, baking quality, and loaf-weight. C. E. MANGELS (Cereal Chem., 1928, 5, 75—77).—The correlation coefficients between water-absorbing capacity and protein content, loaf volume, and loaf weight are given for the years 1923—1926, calculated on the average of the flours from 200—300 samples of hard red spring wheat milled experimentally at the North Dakota Experimental Station. As regards protein and loaf volume, the correlation with water absorption is low, but a coefficient of correlation above 0.50 is found between water absorption and loaf weight for each year studied.

F. E. DAY.

Improvements in the fermentation of bread produced by (the addition of) yeast nutrients. L. ELION (Z. angew. Chem., 1928, 41, 230—231).—The fermentation of dough, as measured by the amount of carbon dioxide evolved, is increased by the addition to the mixing of yeast (1 g.) and flour (50 g.) of small amounts (0.1 g.) of yeast nutrients such as carbamide, peptone, asparagine, and ammonium chloride, tartrate, lactate, or phosphate. Ammonium phosphate gave the most marked increase, pronounced increase being also observed with the lactate. When the proportion of yeast in the mixing was increased to 3 g., the increase in fermentation produced by addition of the nutrients was less pronounced, and a deficiency of sugar was observed. With yeast extract in place of ammonium phosphate fermentation was increased, but sugar deficiency occurred even with 1 g. of yeast, although in the absence of the extract sufficient sugar was always present. Addition of "super-rapidase" produced a remarkable increase in fermentation, and this product appears to act as a sugar producer, by virtue of its diastatic properties, in addition to functioning as a yeast nutrient.

R. BRIGHTMAN.

Determination of chlorine in bleached flour. A. SEIDENBERG (J. Assoc. Off. Agric. Chem., 1928, 11, 132—135).—The flour was thoroughly extracted by

the ordered addition of definite quantities first of 70% and then of 95% alcohol by vol. followed by ethyl ether and light petroleum to avoid the formation of emulsions. The mixed solvents, after separation from the flour, were washed with water, evaporated with alcoholic soda prepared from chlorine-free sodium, and the residue was charred and extracted with dilute nitric acid. The chloride was then determined in the acid solution by means of potassium thiocyanate. Duplicate determinations agreed within 6—7 pts. per million. In all cases the amount of chlorine found was less than that used in bleaching.

F. R. ENNOS.

Determination of field corn in canned mixtures of field and sweet corn. J. L. HEID (J. Assoc. Off. Agric. Chem., 1928, 11, 136—138).—After removal of the liquor and debris by flotation, the kernels are hardened with alcohol and a representative sample of about 100 kernel fragments is taken for examination. The kernels are then cut through, and a portion removed from the centre of each is tested separately with iodine stain on a white tile. A dense brown cloud disseminates from the sweet corn which contains dextrin, whilst the field corn gives the blue-black starch-iodide colour, the proportion of each kind of corn being determined by counting the number of dextrin and non-dextrin containing particles.

F. R. ENNOS.

Feeding trials with milch cows using ammonium acetate as a substitute for protein foods in agricultural practice. H. BAREISS (J. Landw., 1928, 75, 265—324).—In agreement with the work of Pasch it was found possible to replace 25% of the digestible protein in a ration with ammonium acetate with no ill-effect on the cow. Normal live-weight increases in the animals occurred. There was a slight decrease in milk yield, but corresponding increase in the fat content. The utilisation of ammonium acetate by the animal takes place in conjunction with the amides present in the other food material.

A. G. POLLARD.

Large-scale production of yeast as fodder. E. G. STICH (Chem.-Ztg., 1928, 52, 149—150, 170—171).—The possibility of producing yeast economically from molasses as a substitute for other imported food-stuffs is discussed. In order that the price (calculated on the protein content) should be sufficiently low, 100 kg. of sugar must yield 76—80 kg. of dry yeast containing 45% of digestible protein, and this yield is obtainable only by sufficient aeration during the fermentation process. Tables showing the power requirements for this aeration and the costs of production are given, and several alternative factory processes are outlined.

W. J. POWELL.

Volumetric method for determination of proteins in milk. [Mrs.] A. JONESCO-MATIU and C. V. BORDEIANU (Bul. Soc. Chim. România, 1927, 9, 76—85).—A rapid method for the determination of proteins in milk by precipitation in a graduated tube with a 5% solution of mercuric chloride in acetone and measurement of the volume of the precipitate is described. To the mercuric chloride solution (7.5 c.c.) 2.5 c.c. of milk are added drop-wise from a fine pipette, the solution

is well shaken to dissolve out all fats from the precipitate and ultimately left to settle for 24 hrs. at 18°. The volume of the precipitate multiplied by a factor (which varies slightly from tube to tube, being 1.02 in the apparatus used) gives directly the number of grams of protein in 100 c.c. of the sample. The value so obtained never differs from that determined by the Kjeldahl method by more than 2%. The value is also exact for diluted milk containing up to 30% of added water, and when the volume of precipitated protein is less than 2.8 c.c. fraudulent dilution may be assumed. By simultaneous determinations of the density, fat content, and protein content of milk samples it is possible to detect the fraudulent dilution of milk, separation of the cream, or cream separation and dilution combined.

J. W. BAKER.

The sand-Gooch method for analysis of butter. L. C. MITCHELL (J. Assoc. Off. Agric. Chem., 1928, 11, 138; cf. B., 1926, 605).—90-Mesh crystalline alumina (R.R. Alundum, Norton Co., Worcester, Mass., blue label) forms a satisfactory and easily obtainable substitute for the white river sand originally used in this method.

F. R. ENNOS.

Cause of "fishiness" in dairy products. W. L. DAVIES and A. T. R. MATTICK (Nature, 1928, 121, 324).—Butter-fat olein easily absorbs oxygen yielding a labile peroxide, the reaction being strongly catalysed by salts of heavy metals, especially copper. The nitrogenous base portion of lecithin is thereupon oxidised with the production of volatile bases (chiefly trimethylamine) and their fatty acid salts. Appreciable amounts of copper were present in all fishy dairy products. In the absence of metallic contamination the absorption of oxygen and development of rancidity would be slow.

A. A. ELDRIDGE.

Detection and determination of added moisture in sausage. P. A. SIGLER (J. Assoc. Off. Agric. Chem., 1928, 11, 112—125).—The water content of the various kinds of meat used in sausage when calculated on the fat- and ash-free material is nearly constant at about 79%. The ratio of protein (nitrogen content $\times 6.25$) to that of water, calculated on the original meat, is approximately 1 : 4. The percentage of added moisture in sausage may consequently be determined with sufficient accuracy for practical purposes by deducting from the total moisture the quantity of water naturally derived from the ingredient meats, as found by multiplying the protein content by 4.0.

F. R. ENNOS.

PATENTS.

Production of a food product from vegetables containing starch. L. E. JONES, Assee. of H. M. HEIMERDINGER (B.P. 260,277, 21.10.26. U.S., 21.10.25).—Whole vegetables, e.g., potatoes, are boiled with water until the skins are in a filmy condition, and the cooked pulp containing all the nutrient materials is freed from the skins etc. by pressing with a continuous supply of water through perforations in a rigid wall. The mixture of pulp and water is then dried by spraying it in and across a current of air heated at 260—315°, thus yielding

white granules containing 5–8% of water and a high proportion of yeast food substances. F. R. ENNOS.

Production of bread and the like baked goods. L. E. JONES, Assec. of H. M. HEIMERDINGER (B.P. 260,278, 21.10.26. U.S., 21.10.25).—Cereal flour is mixed with the flour-like product obtained as in B.P. 260,277 (preceding) in proportions varying from 0.5–35 pts. of the latter to 100 pts. of the former, and the resulting mixture is leavened, converted into dough, and baked.

F. R. ENNOS.

Manufacture of an edible fat. H. A. NEWTON (B.P. 284,368, 25.9.26).—A vegetable fat is heated with finely-divided onions.

F. R. ENNOS.

Edible fat composition. H. E. DUBIN, Asst. to H. A. METZ LABORATORIES, INC. (U.S.P. 1,656,474, 17.1.28. Appl., 10.10.25).—The composition described in U.S.P. 1,624,164 (B., 1927, 530) is modified by the addition of a fat-soluble vitamin concentrate. T. S. WHEELER.

Separation of liquids. J. ALSBERG, Asst. to B. HELLER & Co. (U.S.P. 1,656,716, 17.1.28. Appl., 31.10.25).—Oily constituents of value as food flavours are separated from the distillate obtained by heating maple or birch wood by saturating it with sodium chloride.

T. S. WHEELER.

Manufacture of an easily-soluble cocoa powder. H. BOLLMANN (U.S.P. 1,660,541, 28.2.28. Appl., 15.12.25. Ger., 30.10.25).—See B.P. 262,239; B., 1927, 171.

Method and apparatus for tempering chocolate. NAT. EQUIPMENT Co., Assecs. of A. L. BAUSMAN (B.P. 271,066, 4.5.27. U.S., 12.5.26).

Manufacture of a food composition [from albumin and sugar]. A. LEO (B.P. 282,923, 18.10.26).

Continuous production of butter. C. G. T. SALENUS (B.P. 284,423, 19.11.26).

Treatment of fresh fruits and vegetables [by spraying]. H. G. ZELLNER (B.P. 284,408, 29.10.26).

Cleaning of milk-stained metal (B.P. 284,778).—See X.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Assay of a so-called cod-liver oil extract for vitamin-A content and calcifying properties compared to cod-liver oil. H. E. MUNSELL and H. BLACK (J. Amer. Pharm. Assoc., 1928, 17, 139–144).—A commercial product described as an alcohol-soluble extract of cod-liver oil has been tested biologically for its content of vitamin-A and -D. It is indicated that 100 mg. of the extract contain not more than one fifth as much vitamin-A as is present in 1 mg. of cod-liver oil, and that the vitamin-D content is not greater than that of cod-liver oil.

E. H. SHARPLES.

Properties and technical application of camphor oils. W. VAUBEL and N. NEDELSCHIEFF (Farben-Ztg., 1928, 33, 1406–1409).—The physical and chemical constants of three commercial light camphor oils have been determined and are compared with those of turpentine oil. The limiting values for the three oils and

those for turpentine oil are d^{18} 0.8589–0.8756, 0.8630, n^{20} 1.4672–1.4788, 1.4700, α –63.25° to –80.4°, +36.45°, boiling range 165–184°, 157–170°, evaporation residue 1.09–2.6, 1.66, bromine value 99.91–160.93, 231.6, flash point 41–43°, 33°, time of evaporation at 20–23°, 10–12 hrs., 10–11 hrs., acid value 0.26–1.6, —. Colour reactions and the drying periods of mixtures of the oils with both linseed oil and varnish show that the camphor oils are quite suitable for use as thinners and solvents in the paint and varnish industry. They have a pleasant odour, and are free from dangerous physiological action after application.

E. H. SHARPLES.

Absolute essence of *Salvia sclarea*. Y. VOLMAR and A. JERMSTAD (Compt. rend., 1928, 186, 517–519).—From the absolute essence of *S. sclarea*, m.p. 35–36°, n_D^{20} 1.5038, d 0.9826, α_D^{20} + 4° 12' (alcoholic solution) were isolated 42% of a crystalline substance *sclareol*, 28% of a sesquiterpene *alcohol*, $C_{15}H_{26}O$, b.p. 169–178°/1 mm., d^{15} 0.9841, n_D^{20} 1.5138, α_D^{20} + 24° 47', 21.8% of volatile constituents (chiefly linalyl acetate and linalool with traces of an unsaturated ester, $C_7H_{12}O_2$), and a small amount of acetic acid and of a free unsaturated acid.

B. W. ANDERSON.

Causes of deterioration of strophanthus seed during storage. E. PICKERING (J. Amer. Pharm. Assoc., 1928, 17, 121–123).—An examination of the causes of deterioration of strophanthus seeds suggested by published work. The seeds should be protected from light, moisture, heat, and inoculation with mould spores.

E. H. SHARPLES.

Standardisation and stabilisation of *Nux vomica*, *Gelsemium*, and *Veratrum*, and the hydrogen-ion concentration factor. IV. E. E. SWANSON and C. C. HARGREAVES (J. Amer. Pharm. Assoc., 1928, 17, 23–27).—Fluid extract of *Nux vomica* suffers no loss in activity, either chemical or physiological, over a period of 10 years, and its therapeutic value is not affected by changes in hydrogen-ion concentration. Chemical and physiological examination of eleven samples of fluid extract of *Gelsemium* prepared in the years 1917–1927 indicated varying degrees of activity and also showed that the two methods do not correlate, the chemical method being unreliable. Changes in hydrogen-ion concentration do not influence the stability of this extract. The chemical method for the assay of fluid extract of *Veratrum* is also unreliable, and the biological method should be employed. This extract seems to require a definite hydrogen-ion concentration to control its deterioration and stabilisation.

E. H. SHARPLES.

Synthetic drug analysis. IX. **Determination of acetylsalicylic acid (aspirin), phenylcinchoninic acid (cinchophen), and caffeine in admixture.** W. O. EMERY (J. Amer. Pharm. Assoc., 1928, 17, 18–22).—In a mixture of aspirin, cinchophen, and caffeine, caffeine is isolated by extraction with chloroform of a solution of the mixture in aqueous sodium carbonate and evaporation of the extract. The residual alkaline solution is treated with iodised potassium iodide solution (Wagner's reagent), which converts the aspirin into

di-iodophenylene oxide, $C_6H_2I_2O$, which is collected on a filter and weighed. The filtrate, on acidification, yields a precipitate of cinchophen periodide ($C_{16}H_{11}NO_2$)₂.HI.I₂, and iodine. The cinchophen can be isolated and weighed by extracting the precipitate with an ether-chloroform mixture and treating with sulphurous acid. In the absence of caffeine, cinchophen may be determined, either alone or admixed with aspirin, by conversion into the periodide in acetic acid solution and titration of the excess of iodine in the filtrate from the precipitate. Representative determinations of many control and commercial samples by the above methods give very satisfactory results. E. H. SHARPLES.

Indian turpentine. PILLAY and SIMONSON.—See XIII.

PATENTS,

Manufacture of phenolic bases of the alkaloid series. E. MERCK CHEM. FABR., Assees. of M. OBERLIN (G.P. 444,587, 18.7.25).—Alkaloids containing alkoxy groups are dealkylated by heating at 120–200° with aluminium chloride alone or in a suitable solvent, methylimino-groups being under these conditions unattacked. Papaveraldine in 1 hr. at 140–150° gives in 84% yield papaveraldoline (sulphate, $C_{32}H_{22}O_{10}N_2 \cdot H_2SO_4 \cdot 6H_2O$), and at 170–175° papaveroline (sulphate, $C_{32}H_{26}O_8N_2 \cdot H_2SO_4 \cdot 10H_2O$); bromopapaveroline (picrate, $+3H_2O$) is similarly prepared. Quinine gives apoquinine, hydroquinine gives hydrocupreine. Laudanosoline (sulphate, $C_{34}H_{38}O_6N_2 \cdot H_2SO_4 \cdot 4H_2O$) and cotarnoline (sulphate, $C_{20}H_{22}O_6N_2 \cdot H_2SO_4 \cdot 2H_2O$) are obtained from laudanosine and cotarnine, respectively. C. HOLLINS.

Pharmaceutical product. F. MÜLLER, Assr. to CHEM. WORKS (FORMERLY SANDOZ) (U.S.P. 1,655,795, 10.1.28. Appl., 18.5.27. Switz., 3.6.26).—*iso*Butylallylbarbituric acid (1 mol.) and 4-dimethylamino-1-phenyl-2:3-dimethyl-5-pyrazolone (1 mol.) combine in methyl alcoholic solution to a compound of therapeutic value. T. S. WHEELER.

Emulsions to be used as shaving creams, anti-septic ointments, or liniments. T. D. KELLY (B.P. 283,711, 13.12.26).

Silicic acid compounds (B.P. 284,450).—See VII. **Glass vessels** (B.P. 284,888).—See VIII. **Cod-liver oil rich in vitamin-A** (B.P. 283,265).—See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Optical and photographic properties of a series of typical cyanine dyes. O. BLOCH and F. M. HAMER (Phot. J., 1928, 68, 21–34).—The chemical constitution, optical absorption, and photographic sensitising properties of the following cyanine dyes are described: *pseudocyanine*, *isocyanine*, *cyanine*, Sensitol Red (2:2'-carbocyanine), dicyanine (of the type 2:4'-carbocyanine), kryptocyanine (4:4'-carbocyanine), thiocyanine, thiocarbocyanine, thiopseudocyanine, thioisocyanine, indocarbocyanine, oxacarbocyanine, erythroapocyanine, xanthroapocyanine, and indopseudocyanine. In the series

pseudocyanine, *isocyanine*, and *cyanine*, and in the carbocyanines, as the 2:2'-linking is replaced by the 2:4'-, and that by the 4:4'-linking, there is a displacement of the absorption maxima towards the red. The shift towards the red corresponds with the lengthening in the carbon chain which connects the two nitrogen atoms. Also the lengthening of the chain which joins the nuclei gives a similar shift towards the red. The shift is also apparent in the case of the pair thiopseudocyanine and thioisocyanine, and in the replacement of the methenyl group by a three-carbon chain in the case of thiocyanine and thiocarbocyanine. With the exception of thiocyanine, every type of cyanine dyestuff, under the conditions described, behaves as a photographic sensitiser. Absorption curves were obtained in the presence of alcohol, water, and gelatin; the nature of the medium has a marked effect on the absorption. Addition of potassium iodide to an aqueous alcoholic solution of dicyanine completely changes the character of its absorption curve. The results throw little light on the relation between absorption and sensitisation. The shift between the maxima of absorption and of sensitisation is towards the red, and this shift becomes greater as the dye sensitises further towards the red. The properties of 1:1'-diethylpseudocyanine iodide, 1:1'-diethyl-3:3':3':3'-tetramethylindocarbocyanine iodide, and 2:2'-diethyloxacarbocyanine iodide (cf. Hamer, A., 1928, 76) are described. W. CLARK.

Action of dyes in fogging reactions. LÜPP-CRAMER (Z. wiss. Phot., 1928, 25, 121–128).—The author has previously shown (Camera [Lucerne], 1924, 3, No. 1) that the fogging action of acids, peroxide, and ozone is favoured by the presence of bromide ions, but that the type of plate used has a marked influence on the results. It is now confirmed that the fogging action of acidified bromide solutions varies markedly with the type of plate, and that the reaction does not take place with orthochromatic plates. The inhibition of the action is due to the dye present. Dyes similarly prevent the fogging action of hydrogen peroxide, phenosafranine, erythrosin, and rhodamine-B being effective. The preventative action decreases in the order of the dyes given. This order is probably bound up with the fact that phenosafranine is least easily washed out of the plate by bathing in water. Desensitisers are not specific in preventing fogging by peroxide, so that the observation that they do prevent fogging can no longer be used as a support for the luminescence theory of fogging by peroxide. Phenosafranine has a specific action in destroying the latent image in presence of acidified bromide. One and the same acid bromide solution can produce a marked fog, and on the same plate can destroy the latent image in presence of phenosafranine. W. CLARK.

Photochemical studies. X. Photographic fog. A. REYCHLER (Bull. Soc. chim. Belg., 1928, 37, 33–39).—The greyish "fog" produced when a photographic plate is developed and then fixed with sodium thiosulphate, without previous exposure to light, is due to a number of black granules. Its appearance may be prevented by immersing the unexposed plate for a few

minutes in a solution of an oxidising agent, 0.1% permanganate, 0.2% chromic acid, or 0.01% ferricyanide, and then treating with acidified sulphite. The negative obtained by subsequent exposure is very clear and brilliant. Mercuric chloride may be used for the same purpose, but in very dilute solution, 0.0025%. Immersion should last only a few seconds and no reducing bath is required. Ordinary fog may also be prevented by preliminary treatment of the plate. The oxidising agents generally used are, at the same time, acid and astringent, so, in order to investigate the cause of the clarifying action, the effect of acids alone and of oxidising agents alone has been investigated. Treatment with acid alone, and subsequent exposure, has a very marked clarifying action on the negative. This effect diminishes with increasing dilution until a concentration is reached at which it is negligible. Such a solution, however, can become an excellent clarifying agent if there is added to it an amount of oxidising agent so small that, if alone, it also would be quite inactive. Anthraquinone- β -sulphonic acid has a marked effect even at very high dilutions, viz., 0.0004*N*, because, besides being an acid, it also becomes a strong oxidising agent on exposure to light. If the plates treated with acidified permanganate or chromic acid are dried before exposure the sulphite bath should be omitted in the preliminary treatment. Sodium thiosulphate is also a very effective clarifying agent. The plate may be immersed in a 30% solution for a few seconds, washed, and exposed to the air for a few minutes. If developed and fixed before exposure a denser fog is formed than without treatment, but after exposure a brilliant negative is obtained. The cause of this action is discussed.

M. S. BURR.

PATENTS.

Photographic plates. SOC. ANON. UNION PHOTOGRAPHIQUE INDUSTRIELLE (ÉTABL. LUMIÈRE ET JOUGLA RÉUNIS) (F.P. 619,395, 1.12.25).—The back of the plate is coated with a layer of cellulose acetate containing a plasticiser and a black aniline dye or lamp-black. After evaporation of the solvent an easily strippable coating remains.

W. CLARK.

Manufacture of films for colour photography. SOC. DU FILM EN COULEURS KELLER-DORIAN (B.P. 261,363, 3.11.26. Fr., 16.11.25).—In a reticulated film for colour photography the width of each reticulation is from 10–40% greater than the width of the image of the colour-selecting screen which it projects on the sensitive surface, for a given aperture of the projecting lens.

W. CLARK.

Films for colour cinematography. R. BERTHON (B.P. 274,837, 28.6.27. Fr., 20.7.26).—A reticulated film for colour cinematography which does not require to be treated with a liquid consists of a support of gelatin, celluloid, cellophane, etc., on one face of which is fixed a layer of celluloid or other plastic material carrying colour-selective, refractive, microscopic elements in relief. The other face of the base is provided with a layer pervious to gases and containing a diazo compound or a mixture of a diazo compound with one or more

phenols, which forms a dark-coloured substance under the action of ammonia etc. when it has not been affected by light (Kalle process).

W. CLARK.

Film images resembling drawings. O. BORNHAUSER (G.P. 445,079, 3.10.26).—Contrasty negatives obtained in the normal way are copied on a film carrying a chromated layer which is coated, before or after developing in water, with a protective layer impervious to water and dyed in an aqueous dye solution. A silvered chromate-containing positive film may be used, development being with a normal developer. The film appears as an outline drawing, consisting of a practically colourless layer of chromium hydroxide, with thin lines and free areas of celluloid.

W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

Explosive properties of lacquer-solvent vapours. RICHARDSON and SUTTON.—See XIII.

XXIII.—SANITATION; WATER PURIFICATION.

Correlation between differential tests for colon bacteria and sanitary quality of water. I. M. LEWIS and E. E. PITTMAN (J. Amer. Water Works' Assoc., 1928, 19, 78–92).—Examination by various methods of colon bacteria isolated from waters of different sanitary quality showed the superiority of the "citrate test"; a combination of indole production and the methyl-red reaction is suggested as the best indicator of sanitary quality of waters.

C. JEPSON.

PATENTS.

Preparation of mouth washes and dentifrices. F. BRÄUNLICH (B.P. 259,942, 28.9.26. Czechoslov., 14.10.25).—To tooth pastes or mouth washes are added sulphonated fats or sulphonated fatty acids, or their alkali or ammonium salts, in quantity sufficient to dissolve any admixed volatile oil or other water-insoluble perfume etc. (e.g., 10 pts. of alkali sulphonate to 90 pts. of paste).

B. FULLMAN.

Centrifugal apparatus for the purification of liquids [water]. F. JENSEN, Assr. to B. E. HILL (U.S.P. 1,654,812, 3.1.28. Appl., 21.11.24).—Centrifugal separation of objectionable matter from water to be used for the production of raw ice is effected by cutting the water rapidly while whirling it in opposite directions alternately.

H. HOLMES.

Stabilisation of ferruginous water. C. PEETERS (U.S.P. 1,656,232, 17.1.28. Appl., 8.9.25. Belg., 9.9.24).—The separation of iron from water containing it is inhibited by the addition of a polyhydric alcohol, e.g., glycerol, or of an aldehyde, e.g., dextrose.

T. S. WHEELER.

Water-softening apparatus for use with steam generators or other plant requiring softened water. J. MILLS & Co. [ENGINEERS], LTD., and W. H. PORTER (B.P. 285,629, 20.1.27).

Grading of mixed materials (B.P. 283,645).—See I. Glauconite (U.S.P. 1,657,700).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

APRIL 27, 1928.

I.—GENERAL; PLANT; MACHINERY.

Adhesion. II. (Sir) W. B. HARDY and (Miss) M. E. NOTTAGE (Proc. Roy. Soc., 1928, A, 118, 209—229).—Measurements of the tensile strength of a solid joint between a steel or copper cylinder and plate are described. The joint was formed by melting solid paraffins, fatty acids, or alcohols on the plate, allowing some of the liquid to be drawn under the cylinder by capillary attraction, and then freezing it. A microscopical examination of the disc of "lubricant" found on either the plate or cylinder on breaking the joint supports the view that it is composed of a central plate of flat crystals between two primary layers, one on each of the enclosing solids. The break appears to occur at one or both of the surfaces between the primary film and the plate of crystals. The effect of added impurity on the strength of the joint and the position of the surface of break was investigated, and the relation of the adhesion to the temperature, pressure, chemical composition of the lubricant, and composition of the cylinder and plate is discussed. As in the case of static friction, the adhesion varies linearly with the mol. wt. of the lubricant for a given chemical series. The distance between the surfaces of the cylinder and plate, measured by means of a specially constructed spherometer, was found to be independent, within the limits of error, of the nature of the lubricant and of the enclosing solids. A few measurements are recorded of the tangential pull required to break a joint. The fracture occurs at the same surface as with a normal force. L. L. BIRCUMSHAW.

Effect of thermometric lag in fractional distillation. W. R. ORMANDY and E. C. CRAVEN (J. Inst. Petroleum Tech., 1928, 14, 94—105).—Using mercury thermometers in distillations from a flask fitted with an eight-bulb Young "evaporator" still-head, it was found that a constant, R , called the "speed" of the thermometer, representing the number of degrees the thermometer would rise per second if the bath were maintained always 1° F. above the thermometer reading, depends on the diameter of the bulb and the thickness of its walls, and for the same thermometer depends on the latent heat of condensation of the vapour and the speed at which it passes the bulb. In a liquid bath, R depends on the rate of stirring. In general, the initial lag of the mercurial thermometer depends on the thickness of the layer of condensed liquid on the bulb, or, in the case of a liquid bath, on the layer of cooled liquid around the bulb. Measurements showed that the thickness of film required to produce the observed lag is of the order of 0.25 mm. In accurate distillations the use of a thermocouple is preferable. H. S. GARLICK.

Steam still for volatile acids. D. H. CAMERON (J. Amer. Leather Chem. Assoc., 1928, 23, 42—45).—

A Hortvet tube (J. Ind. Eng. Chem., 1909, 1, 31) is permanently mounted in a glass steam jacket by means of a rubber stopper, into which is also fitted a tube for a steam vent. Discharge of steam from the jacket and through this tube is regulated by means of a piece of rubber tubing and screw clip fitted over the end of the vent tube. Steam is generated from distilled water, free from carbon dioxide, in a 500 c.c. extraction flask, which is fitted to the lower end of the steam jacket. The Hortvet tube is fitted with a Kjeldahl bulb-form spray-trap, and a hole is blown in the vertical tube just below the stopper in the Hortvet tube. 10 c.c. of the liquor under examination are pipetted into the Hortvet tube, the apparatus is assembled, and the Kjeldahl delivery tube fitted to a condenser, and the distillate collected in 0.1N-alkali. D. WOODROFFE.

Technical reaction vessel. E. HOCHBERGER (Chem.-Ztg., 1928, 52, 130—131).—An apparatus suitable for reactions between solids and liquids with evolution of gas consists of a rotating drum mounted with the axis horizontal and provided with a tightly fitting lid on the cylindrical surface. The bearings at both ends are mounted on hollow shafts, which may permit of connexion to two concentric pipes or one single pipe to allow of introduction of liquids or gases and escape of a gaseous product etc. The whole may be made sufficiently gastight to permit of reactions being carried out under pressure. S. I. LEVY.

PATENTS.

Coal-dust furnaces. SIEMENS-SCHUCKERTWERKE G.M.B.H. (B.P. 282,333, 26.4.27. Ger., 17.12.26. Addn. to B.P. 270,295; B., 1928, 215).—In modification of the prior patent, the secondary jets are so numerous that their action on the flame may be considered as continuous; their direction of entry gradually approaches the axis of the flame and their velocity gradually decreases in the general direction of the flame, which is usually downwards. B. M. VENABLES.

Sublimation apparatus. C. A. LYFORD, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,662,070, 13.3.28. Appl., 2.4.20).—Means are provided for introducing steam both above and below the material to be sublimed, placed in a container, and for condensing sublimed material. J. S. G. THOMAS.

Drying of crystals. H. EBEL (G.P. 445,821, 1.4.25).—The crystals at the bottom of the crystallising pan are sucked into a closed vacuum filter until they reach a definite height, when the cover is removed and the mother-liquor passed through the filter and sucked into a collecting vessel, whence after removal of the vacuum it falls by gravity back to the crystallising pan. The vacuum filter is provided with a three-way cock

making connexion with the trough of the crystallising pan and with the vacuum pump. A. R. POWELL.

Recovery of volatile solvents by means of inert gases. R. OERTEL (G.P. 445,564, 26.7.25. Addn. to G.P. 444,913; B., 1928, 111).—The gas carrying the vapour of the solvent is passed from the evaporating chamber through a secondary circuit containing washing vessels and filters to remove oxygen. A. R. POWELL.

Hydrogen-ion comparator. A. HYUGA (U.S.P. 1,659,529, 14.2.28. Appl., 19.4.26).—The colour of light passed through tubes of untreated standard solution and of solution under test treated with an indicator, is compared with that of light passed through standard solution treated with the indicator and untreated test solution. T. S. WHEELER.

Colloidal dispersion of solids. S. CABOT, Assr. to S. CABOT, INC. (U.S.P. 1,662,999, 20.3.28. Appl., 31.1.25. Renewed 28.7.27).—See B.P. 246,874; B., 1927, 800.

Filtering apparatus. J. B. VERNAY (U.S.P. 1,662,726, 13.3.28. Appl., 16.2.27. Fr., 24.2.26).—See B.P. 266,719; B., 1927, 801.

Apparatus for concentrating aqueous liquids. P. J. DE S. A. MEYNARDIE (U.S.P. 1,661,489, 6.3.28. Appl., 6.4.25. Fr., 16.4.24).—See B.P. 232,589; B., 1926, 31.

Gas-analysing apparatus. O. RODHE, Assr. to SVENSKA AKTIEBOLAGET MONO (U.S.P. 1,661,627, 6.3.28. Appl., 19.8.26. Ger., 28.8.25).—See B.P. 257,609; B., 1927, 592.

Furnace or combustion chamber walls. ÉTABL. F. LABESSE (B.P. 280,561, 2.5.27. Fr., 4.5.26).

Process and apparatus for extinguishing fires. EXCELSIOR FEUERLÖSCHGERÄTE A.-G., and H. BURMEISTER (B.P. 285,934, 19.10.26).

[Nozzle for] spraying liquids. BABCOCK & WILCOX, LTD. From BABCOCK & WILCOX Co. (B.P. 286,114, 19.4.27.)

II.—FUEL; GAS; TAR; MINERAL OILS.

Banded coal. H. WINTER (Fuel, 1928, 7, 52—63; Glückauf, 1927, 63, 14).—The work of previous investigators on banded bituminous coal is summarised and compared. It is concluded that fusain and durain must be regarded as well-defined petrological constituents of coal, which is much less the case for vitrain and clarain, together forming the "bright" coal. As a result of the discovery of plant structure in vitrain, the Stopes definition (B., 1919, 452 A), which was based essentially on the absence of structure in this ingredient, has become untenable. A. B. MANNING.

Replaceable bases in the roofs of bituminous coal seams of carboniferous age. E. MCK. TAYLOR (Fuel, 1928, 7, 66—71).—The p_H values and amounts of the replaceable bases present in a large number of rocks forming the roofs of coal seams in Great Britain have been determined. The majority are alkaline in reaction and have the replaceable sodium considerably in excess of the replaceable calcium. Where the rocks differ from this general type there is evidence that the original roof has been removed by denudation and the

present one deposited subsequently. It is concluded that the roofs of these bituminous coal seams have been deposited under such conditions that, either during or subsequent to deposition, they have been in contact with solutions of sodium chloride, and that the base-exchange product has been subjected to hydrolysis in fresh water, yielding an alkaline clay which formed the roof of the seam during coal formation. Such an alkaline medium existing under anaerobic conditions would favour bacterial decomposition of organic matter (cf. B., 1927, 691). A. B. MANNING.

Dry-cleaning of coal for carbonisation. C. W. H. HOLMES (Gas World, 1928, 88, Coking Sect., 32—38; cf. B., 1927, 641).—The influence of the relationship between density, particle size, and ash content on the theoretical possibilities of coal cleaning is discussed with reference to typical coking slacks. Pyritic sulphur is generally readily removable by cleaning, but organic sulphur is not. Representative layouts for the treatment of coking slacks by pneumatic separation are briefly described, and discussed with particular reference to the necessity for sizing, the removal and utilisation of the dust, the effect of moisture in the coal, and the running costs of the process. A. B. MANNING.

Influence of various factors on the products of the carbonisation of coal. H. H. THOMAS (J.S.C.I., 1928, 47, 77—83 T).—Increasing the temperature of carbonisation caused an increase in the yield of gas of reduced calorific value. At 950° the yield per ton was 11,110 cb. ft. of 649 B.Th.U. per cb. ft., and at 1000°, 13,340 cb. ft. at 590 B.Th.U., the net result being an increase in the gaseous therm yield from 72.1 therms per ton at 950° to 78.7 therms at 1000°. These results are presumed to be due to the increased degradation of the tar vapours at the higher temperatures. With the coal screened to pass 1 in. mesh and remain on $\frac{3}{4}$ in. mesh, 11,200 cb. ft. of gas per ton, of 635 B.Th.U. per cub. ft., were obtained (=71.1 therms per ton). By reducing the size of particle the gas yield was increased so that with a particle size through $\frac{1}{4}$ in. on $\frac{1}{8}$ in. 12,000 cb. ft. of gas (616 B.Th.U.) were obtained, equivalent to 73.9 therms per ton. Further results show the effect of the addition of inert material (coke and non-coking coal) to the coal before carbonisation, the object being to obtain a more reactive and combustible coke. Considerable importance is attached to the necessity for the fine grinding of the coals forming a blend, in order that intimate mixing and an even structure in the resultant coke be obtained. The addition of 20% of coke gave an increased yield of gas of reduced calorific value, the therms per ton of coal being the same as obtained from the coal carbonised alone. The reactivity of the coke from the mixture was only slightly increased. A blend of 70% of coking gas slack with 30% of a highly oxygenated non-coking coal gave a coke of considerably increased reactivity. The thermal yield from the blend was approximately equivalent to the sum of the yields of the two coals when carbonised separately. The rate of carbonisation in this case was not greatly increased on that of the gas slack alone; this may result in the output of a given plant being considerably reduced as the non-coking coal has only a low gas-making value.

It is suggested that the charge be removed from the retort before carbonisation is complete, after which time no appreciable loss of gaseous therms would occur. In this way the output of the plant could be maintained and the benefit of the increased reactivity in the coke obtained. The experiments were carried out in a small 1/1000-ton test plant which is described.

Low-temperature distillation of coal briquettes. P. WEISS (Chim. et Ind., 1928, 19, 195—204).—The solid fuel obtained by carbonising briquetted coal at low temperatures varies with the type of coal used, the rate of heating, the fineness of the coal, the pressure to which the briquettes have been submitted, and the proportion of binder. The influence of these factors has been investigated, the carbonisations being carried out in an internally-heated retort in which the heating medium passes successively through a number of chambers, its point of entry being moved periodically from one chamber to the next so that the contents of each chamber in turn are subjected to higher and higher temperatures until carbonisation is complete. Briquettes made from anthracitic coals with the correct proportion of pitch as binder can be satisfactorily carbonised at a rate of heating of 3° or more per min., in either internally- or externally-heated retorts. Swelling coals require a careful regulation of the rate of heating, especially over certain critical temperature ranges, when the rate of heating may have to be reduced below 0.1° per min. in order to produce briquettes of the required density and strength. With a suitable rate of heating a variation in the amount of pitch from 6 to 8% makes little difference to the briquettes. The density and strength of the carbonised briquettes increase at first with the pressure under which the briquettes are formed, pass through a maximum, and then fall off; the position of the maximum, however, depends on the rate of heating employed, which should therefore be so chosen that the maximum is just reached for the working pressure used. In general, better briquettes are obtained if the coal is more finely ground, but there is evidence of an optimum particle size for each coal. For non-coking coals and lignites a binder containing relatively light oils is necessary; the strength of the briquettes is markedly increased by the addition of a small proportion of finely-powdered sulphur to the mixture of coal and binder.

A. B. MANNING.

Combustion of powdered coal. B. MOORE (Trans. Inst. Chem. Eng., Advance Proofs, March, 1928, 12—24).—The degree of fineness of powdered coal affects considerably its rate of combustion, ignition properties, and "combustible capacity," as determined by the method previously described (B., 1925, 486; 1927, 130). The rate of combustion increases and the "glow point temperature" falls with increasing degree of fineness of the coal. The results indicate that there is a critical temperature and a critical degree of fineness at which the combustion of the particles becomes almost instantaneous.

A. B. MANNING.

Natural bitumens, with special reference to bituminous shale. W. ZWIEG (Petroleum, 1928, 24, 243—246).—In parts of north Germany a type of peat

resembling cannel coal is found which contains considerable quantities of bituminous substances. Whilst ordinary peat is classed with brown coal, this shale-peat is more suitably grouped with oil-shale; in the first class the hydrogen content per 100 pts. of carbon varies from 8.3 to 2.1, but in the second the ratio is constant at 12 pts. of hydrogen. The dry, ash-free peat contains 82.7% of volatile matter, being almost identical in this respect with an Australian shale; the yield of dry bitumen obtained on distillation is 25% by wt. Gas-making tests carried out by the Geipert method are compared with those given by carbonisation of Australian shale in horizontal retorts; 52.6% of the potential heat in the peat can be obtained in the gas.

R. H. GRIFFITH.

Preparation, purification, and analysis of pyro-ligneous oils. R. HUERRE (J. Pharm. Chim., 1928, [viii], 7, 58—69).—A description and discussion of the treatment and analysis of oils obtained by the dry distillation of various woods, particularly with regard to the nature and recovery of the phenolic substances.

E. H. SHARPLES.

Relation of the micelle to the medium in asphalt. F. J. NELLENSTEYN (J. Inst. Petroleum Tech., 1928, 14, 134—138).—Asphalt consists of a medium and a disperse phase, the asphalt micelle containing a lyophile part (protective substances) and a lyophobe part (elementary carbon). Flocculative and peptising reactions cause changes in the stability of the system depending on the relation between the micelles and the oily medium. Interfacial tension of the micelle is changed by adding a liquid which changes the surface tension of the medium. Of liquids miscible with the asphalt medium those of low surface tension are flocculating reagents, whereas those possessing high surface tension are peptising reagents. The higher the surface tension the better is the solvent power. The surface tensions of petroleum residues and blown asphalts range from 23.1—25.2 dynes/cm., and for coal tar 32.0 dynes/cm. Precipitation is never complete with liquids of low surface tension, e.g., benzene. Only by adding a liquid not completely miscible with the medium or the protective substances, such as ether-alcohol mixture, can this be achieved, but true flocculation no longer occurs and the "soft asphalt" obtained contains a large proportion of oily compounds.

H. S. GARLICK.

Diesel-engine performance on oils obtained from the low-temperature carbonisation of coal. J. S. BROWN (J. Roy. Tech. Coll. Glasgow, 1927, [4], 76—84).—The performance of a Diesel engine when three oils obtained from coal by low-temperature carbonisation processes are used as fuel has been investigated. In each case the engine was worked up to full load and normal jacket temperature on a normal petroleum oil, and the fuel was then gradually changed to that under test. In one case excessive viscosity caused an air-lock in the feed, but the performance on the other two oils was entirely satisfactory with the engine under full load. In the case of one oil only was it possible to avoid pre-ignition when the engine was running at no load. In neither case was it possible to start the engine without preheating the jackets. Two of the three oils gave a small amount of deposit when mixed with Shell-

Mex Diesel grade oil. These oils are preferable to the creosote oils previously obtained from coal, and it is considered that a little further improvement in the quality would give them all the properties of a petroleum oil. L. M. CLARK.

Spreading of lubricants on solid surfaces. P. WOOG (Compt. rend., 1928, 186, 71—73).—The spreading properties of typical animal, vegetable, and saturated neutral mineral oils on various metallic surfaces have been investigated at 100°. Whilst the animal and vegetable oils spread rapidly at low temperatures over all the surfaces tested, the mineral oil (d_{15}^4 0.8647, η (abs.) 0.0785 at 50°, and 0.0024 at 100°) usually required a higher temperature and a coating of stearic acid over the metallic surface in order to produce rapid spreading. The stearic acid neutralises the surface by the production of a layer of active oriented molecules. The phenomenon is most marked for cold-beaten surfaces. J. GRANT.

Regeneration of used [lubricating and insulating] oils. K. TYPKE (Gas- u. Wasserfach, 1927, 70, 1289—1291).—The removal of suspended matter from used oils is not sufficient purification, and treatment with fuller's earth is adequate only for slightly altered oils. Stirring with sulphuric acid and, in some cases, with sodium hydroxide is necessary, and may, with advantage, be followed by treatment with fuller's earth (stirring with powdered earth and filtering, or allowing the oil to percolate through a bed of granular earth). Used oils containing light constituents of low flash point should not be mixed with oils of high flash point, otherwise the former must be removed, by some method of evaporation or distillation, prior to refining. The regenerated oils should satisfy the same requirements as a fresh oil, and an important test of the adequacy of regeneration is to note the extent of tar formation when oxygen is passed for 7 hrs. through the oil at 120°.

W. T. K. BRAUNHOLTZ.

Petrol engine lubricants and lubrication. C. I. KELLY (J. Inst. Petroleum Tech., 1928, 14, 115—133).—A general discussion of the theory of lubrication and its application to automobile conditions.

H. S. GARLICK.

Liquid fuels other than petroleum. A. E. DUNSTAN and H. G. SHATWELL (J. Inst. Petroleum Tech., 1928, 14, 64—77).—A summary of contemporary information regarding the production and properties of motor fuel derived from the low-temperature carbonisation and hydrogenation of coal, and sundry gas reactions resulting in methyl alcohol, synthol, and synthetic petroleum.

H. S. GARLICK.

Blasting in tunnels. GARDNER and others.—See XXII.

PATENTS.

Ovens for treating fuels or fuel-containing materials by heat. O. HELLMANN (B.P. 272,493, 1.6.27. Ger., 8.6.26).—An oven for the heat treatment of fuels has a movable annular platform carrying transverse, evenly spaced heating chambers, between which are spaces open towards the edges of the platform and designed to receive the raw material. The platform and heating chambers are enclosed within stationary walls, inclined at the natural angle of repose of the material,

and provided with opposite openings at one point through which the product is discharged by a pusher head.

A. B. MANNING.

Activation of carbon or carbonaceous substances with simultaneous production of combustible gases. SOC. DE RECHERCHES & D'EXPLOIT. PETROLI-FÈRES (B.P. 269,477, 23.11.26. Fr., 15.4.26).—Carbon to be activated, or carbonaceous material to be converted into active charcoal, is enclosed in chambers with walls which are permeable to gas, and is subjected to the action at high temperatures of gases containing no free oxygen, e.g., combustion gases rich in carbon dioxide and steam, which reach the material by diffusion through the walls. The speed of activation is controlled by regulating the gaseous pressure within the chambers. The combustible gases formed in the chambers are withdrawn and used for heating purposes. The chambers or retorts are preferably of a shape, e.g., narrow and flat, which possesses a large surface area. A. B. MANNING.

Drying material [coal] by means of fumes evolved in a subsequent operation. KOHLENVEREDLUNG A.-G. (G.P. 446,042, 19.11.22).—In the drying of wet substances, such as coal, the heat of the fumes or smoke evolved in the last stages of the drying operation is utilised by passing the gases through a secondary dryer arranged before or after the main dryer. Previous to passing to this apparatus the fumes may be superheated by passing them over glowing material to cool it below the ignition point.

A. R. POWELL.

[Smokeless] fuel. T. NAGEL (U.S.P. 1,658,814, 14.2.28. Appl., 15.11.26).—A low-volatile bituminous coal is mixed with a binder of the type described in U.S.P. 1,507,675—6 (B., 1924, 1003), and the mixture is briquetted and heated at 400°.

T. S. WHEELER.

Utilisation of subdivided coal. N. TESTRUP, O. SODERLUND, T. GRAM, and TECHNO-CHEMICAL LABORATORIES, LTD. (B.P. 284,405, 28.10.26).—To coal slurry containing a high percentage of water, dried powdered coal is added in such proportion that a friable mixture is produced, and the latter is then dried in a current of air, the water content being reduced until the powder will flow easily, but at the same time retain sufficient water to prevent the formation of dust. The drying may be continued further, but in that case the dust-laden air is conveyed to a furnace where the dust is burnt, or the dust may be recovered by suitable means.

A. B. MANNING.

Apparatus for the production of carbon black. W. F. FULTON, Assr. to PALMER CORP. (U.S.P. 1,658,676, 7.2.28. Appl., 31.5.27).—Apparatus for the production of carbon black comprises a rotary collector cylinder communicating with a gas supply and for heating the gas, manifolds for receiving the hot gas and delivering it to passages in arches between which a horizontal combustion tube designed to direct decomposed gas substantially normally against the rotary collector is arranged.

J. S. G. THOMAS.

Preparation of carbon black. HARTSTOFF-METALL A.-G. (HAMETAG), Assecs. of E. PODSZUS (G.P. 445,498, 1.10.24).—For the grinding of carbon to colloidal fineness a mill with steel balls of 1—2 mm. diameter is used. The carbon powder mixed with the balls may be subjected

to a high-speed counter-current of gas or air to increase the efficiency of the grinding. A very even product with large surface is obtained.

C. HOLLINS.

Production of liquid hydrocarbons and other products by the destructive hydrogenation of coal, tars, and like carbonaceous materials. I. G. FARBENIND. A.-G. (B.P. 256,965, 10.8.26. Ger., 14.8.25).—Coal, tars, etc. are converted into valuable liquid hydrocarbons by being submitted, in a gaseous or finely-divided state, to the action of hydrogen or gases containing or giving rise to hydrogen, under a pressure of at least 50 atm. and at high temperatures, in the presence or absence of catalysts. The gases may be recirculated for the treatment of fresh material, and a mixture of hydrogen and water vapour or water vapour alone added to make up for the hydrogen consumed. No coke is produced in the process, and the rate of throughput is higher than when working without atomising the material.

A. B. MANNING.

Manufacture of liquid and other hydrocarbons and derivatives thereof by the destructive hydrogenation of coal, tars, etc. I. G. FARBENIND. A.-G. (B.P. 257,910, 12.8.26. Ger., 2.9.25).—Coal, tars, or other carbonaceous materials are hydrogenated under pressures of at least 50 atm. and at high temperatures in a current of the hydrogenating gases; the starting material is employed in a moist state, or water may be added during the process. The formation of coke is avoided, and the process can be carried out continuously so that the material is practically completely consumed.

A. B. MANNING.

Manufacture of liquid and other hydrocarbons and derivatives thereof by the destructive hydrogenation of tars, mineral oils, resins, asphalts, etc. I. G. FARBENIND. A.-G. (B.P. 282,814, 10.8.26. Ger., 14.8.25. Addn. to B.P. 256,964; B., 1928, 221).—The procedure of the main patent has been extended to the hydrogenation of liquids or materials which are liquid under the working conditions; the addition of 5–30% of brown coal or peat to the starting material facilitates the hydrogenation and prevents coking.

A. B. MANNING.

Manufacture of unsaturated gaseous hydrocarbons and mixtures of carbon monoxide and hydrogen. I. G. FARBENIND. A.-G. (B.P. 269,547, 12.4.27. Ger., 15.4.26).—Gas mixtures containing saturated hydrocarbons are subjected to a process, e.g., passage through an electric arc, or incomplete combustion, partially converting them into unsaturated hydrocarbons. These are removed by some suitable process (acetylene, for example, by conversion into benzene, and absorption of the latter in active charcoal), and the residual gas is then treated with steam or oxygen or both, either in the presence or absence of catalysts, to give a mixture of hydrogen and carbon monoxide.

A. B. MANNING.

Conversion of heavy hydrocarbons into light and stable hydrocarbons. P. GRÉNÉ (B.P. 285,668, 21.3.27).—Tars, crude oils, etc. are cracked and converted into hydrogenated products by passage through heated tubes containing layers of different catalysts. The oils to be treated are subjected to a preliminary

mild cracking process, wherein coke and gas production is reduced to a minimum, and the vapours are diluted with a constant concentration of suitable neutral gases, in opposition to the production of gaseous products, before passage through the heated catalyst tubes. These contain (1) a porous carbonaceous substance, such as coke, which promotes cracking, prevents carbon formation, and promotes the decomposition of organic sulphur compounds; (2) a layer of manganese dioxide which removes sulphur compounds, and may be regenerated by means of an air blast; (3) a further layer of a carbonaceous catalyst capable of causing hydrogen to be generated from the water vapour present; and (4) a hydrogenation catalyst such as reduced nickel.

C. O. HARVEY.

Distillation of hydrocarbons, and fuel produced thereby. L. T. BATES (U.S.P. 1,657,815, 31.1.28. Appl., 5.6.26).—Coal, mixed with liquid hydrocarbons, is pulverised at a temperature sufficiently high to vaporise the volatile matter present in the mixture.

C. O. HARVEY.

Controlling the destructive distillation of petroleum oil. R. W. HANNA, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,658,241, 7.2.28. Appl., 12.2.23).—The oil is cracked and vaporised and the evolved vapours are passed (under the pressure of the cracking reaction) through a three-stage separating system. In the first stage the vapours are scrubbed with a descending shower of the original feed oil mixed with the reflux descending from the second stage; in the second stage the reflux condensate from the third stage is used as the scrubbing liquid, and in the third the vapours are cooled to the desired discharge temperature by means of a liquid other than that undergoing treatment. The combined reflux condensates, admixed with feed oil, are passed to the cracking chamber.

C. O. HARVEY.

Oil-shale distillation apparatus. C. D. TRIPP (U.S.P. 1,658,143, 7.2.28. Appl., 12.7.24).—The shale is introduced at one end of a horizontal retort, the bottom of which is covered with molten metal, and the other end of which is fitted with an inverted weir dipping below the surface of the metal bath. Means are provided for mechanically propelling the shale towards the weir, and for delivering the gangue beneath the weir to a point of discharge. The products of distillation pass out from the upper portion of the retort.

C. O. HARVEY.

Treatment of hydrocarbon oils. H. W. CAMP, Assr. to DOHERTY RES. Co. (U.S.P. 1,657,524, 31.1.28. Appl., 29.11.22).—The temperature gradient in a dephlegmating column is controlled by allowing a part of the chilled condensate to flow from a point below the top of the column in a downward direction, the vapours from the still passing upwards. The other portion of the condensate from the column is mixed with a further quantity of the original oil and injected into the still.

C. O. HARVEY.

Treatment of [hydrocarbon] oils. R. J. DEARBORN, Assr. to TEXAS Co. (U.S.P. 1,658,077, 7.2.28. Appl., 18.4.22).—In a process for the production of gasoline, a continuous stream of oil is raised, by passage through a tubular still, to a temperature adequate only for cracking when aluminium chloride is present. The

oil without further heating comes in contact with aluminium chloride in a separate reaction chamber, and the vapours produced are partially condensed, the condensate passing back to the still for recirculation.

C. O. HARVEY.

Apparatus for treating oils. G. D. SIMS, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,658,420, 7.2.28. Appl., 17.6.21. Renewed 10.3.27).—The level of the oil in a vaporising chamber which is connected with a pressure still is indicated by means of a gauge which communicates with the base of the chamber via a cooling device. The vapour space above the oil in the gauge communicates with the receiver for condensate, whereby equal pressure is maintained on the oil in the gauge and that in the chamber.

C. O. HARVEY.

Preparation of hydrogen and hydrogenation of hydrocarbons. A. MALEVILLE (F.P. 621,902, 23.9.26).—Molten metals, *e.g.*, zinc, and water are used. The metallic oxides formed must be capable of reduction by carbon below a red heat. The hydrogen so produced is allowed to act on the coal or hydrocarbons in the bath itself.

A. B. MANNING.

Extraction of petroleum products from bituminous material. P. DVORKOVITZ (B.P. 285,564, 17.11.26).—The yield of the products obtained by the destructive distillation of bituminous materials (*e.g.*, coal) is augmented by introducing into the charged retort, preheated to a temperature (500°) sufficiently high to effect the removal of a portion of the organic matter, a solvent in liquid or vapour form capable of removing a further portion of the organic matter. The solvent (*e.g.*, petroleum or gas tar) is subsequently recovered for re-use.

C. O. HARVEY.

Oil composition suitable as a power-transmitting agent in hydraulic brakes for automobiles etc. E. A. EVANS, and C. C. WAKEFIELD & Co., LTD. (B.P. 285,144, 10.11.26).—Castor oil is mixed with an alcohol or mixture of alcohols of mol. wt. higher than that of ethyl alcohol, *e.g.*, benzyl alcohol and cyclohexanol.

A. B. MANNING.

Lubricating composition. E. A. NILL, Assr. to F. C. ACKER and J. R. DALL (U.S.P. 1,659,149, 14.2.28. Appl., 4.5.23. Cf. U.S.P. 1,659,150; B., 1928, 293).—A mixture of a mineral oil and a fatty-acid anilide, *e.g.*, stearanilide, is claimed.

T. S. WHEELER.

Reclamation of used crank-case oil. W. H. ELLERINGTON (U.S.P. 1,662,701, 13.3.28. Appl., 23.1.26).—Fine particles are removed from lubricating oil by intimately mixing the latter with soap, sodium silicate, and talc.

H. ROYAL-DAWSON.

Use of heavy oils in internal-combustion engines. SOC. ANON. LE CARBONE (B.P. 262,407, 10.11.26. Fr., 5.12.25).—The mixture of air and atomised fuel is subjected to the action of active carbon at 300–400° immediately before admission to the engine, a part of the fuel being thereby transformed into light oil vapours. The catalyst, which may either be interposed between the carburettor and the engine inlet or contained in the carburettor itself, consists of a block of activated wood charcoal preferably provided with oblique passages and so mounted that the proportions of air and fuel mixture which pass respectively through the body of the charcoal

and through the passages can be varied to suit the running of the engine. The tube containing the charcoal block is jacketed so that by the use of a suitable heating medium its temperature can be maintained above 250°.

A. B. MANNING.

Prevention of pinking or knock in internal-combustion engines. ASIATIC PETROLEUM CO., LTD., and A. C. EGERTON (B.P. 279,560, 29.7.26).—Thallium vapour or the vapour of a thallium compound, *e.g.*, thallium ethyl, ethoxide, oleate, etc., is introduced into the cylinder.

H. ROYAL-DAWSON.

Bonding of activated carbon. A. B. RAY, Assr. to CARBIDE & CARBON CHEMICALS CORP. (Re-issue 16,896, 6.3.28, of U.S.P. 1,565,328, 15.12.25).—See B., 1926, 119.

Dry-cooling of coke. R. WUSSOW (U.S.P. 1,661,211, 6.3.28. Appl., 19.10.26. Ger., 17.1.23).—See B.P. 251,530; B., 1926, 573.

Prevention of [coal] mine explosions. W. E. TRENT, Assr. to TRENT PROCESS CORP. (Re-issue 16,914, 20.3.28, of U.S.P. 1,642,912, 20.9.27).—See B., 1927, 900.

Liquid fuel. E. M. FLORES (U.S.P. 1,657,961, 31.1.28. Appl., 9.6.22. Argentine, 16.1.22).—See B.P. 249,577; B., 1926, 524.

Coke oven closures. H. J. LIMBERG (B.P. 276,005, 11.8.27. Ger., 11.8.26).

Machines for separating coal and for other analogous purposes. W. H. BARKER (B.P. 286,452, 1.3. and 28.6.27).

Coke quenching and loading appliances. Dr. C. OTTO & COMP., G.M.B.H. (B.P. 280,944, 18.11.27. Ger., 22.11.26).

[Automatic] apparatus for separating benzol from waste-water. W. LINNMAN, JUN. (B.P. 272,512, 8.6.27. Ger., 8.6.26).

Water-gas (B.P. 282,573).—See VII. **Paving mixtures** (U.S.P. 1,662,377). **Road binder** (U.S.P. 1,662,299).—See IX. **Resin from tar oil** (U.S.P. 1,658,281).—See XIII.

III.—ORGANIC INTERMEDIATES.

Vanillin from sulphite[*-*cellulose] waste liquors. K. KÜRSCHNER (J. pr. Chem., 1928, [ii], 118, 238–262).—The evidence from sublimation experiments in favour of the view that pine lignin is a colloidal coniferin complex (cf. A., 1925, 890, 1387; Routala and Sevón, "Über die Ligninstoffe") is reviewed. Considerable amounts of vanillin are formed when a rapid current of air is passed through boiling alkaline sulphite-cellulose waste liquors (cf. Grafe, A., 1905, i, 22). Using a neutralised liquor containing 64% of total solids and 24.8% of lignin, the optimum alkalinity, concentration, and reaction period are obtained when 10 c.c. are refluxed with 10 g. of sodium hydroxide or 10–20 g. of potassium hydroxide and 30 c.c. of water for 1.5 hrs. After neutralisation and filtration the vanillin (0.53 g. = 21–22% of the lignin) is determined colorimetrically in both solution and precipitate using phloroglucinol and hydrochloric acid or orcinol and sulphuric acid. This yield,

which is in good agreement with the proportion of aromatic derivatives obtained by other degradative methods (cf. Klason, A., 1923, i, 187; Heuser, A., 1923, i, 900), is not improved by the use of oxygen nor by adding copper dust, mercuric, cupric, or nickelic oxides, ferric chloride, or potassium nitrate, and is decreased by adding sodium peroxide or potassium ferriocyanide. Extraction of the solution and the dried precipitate with ether affords 0.5—1.0 g. of crude vanillin. This is obtained crystalline by sublimation and then contains guaiacol but no pyrocatechol or oxalic acid. Experiments with decayed pine-wood and lignite are also described.

H. E. F. NOTTON.

Chemical treatment of trade waste. IV. Waste from organic ester synthesis. F. D. SNELL and C. T. SNELL (Ind. Eng. Chem., 1928, 20, 240—241).—The wastes to be treated consist of a calcium sulphate sludge from the stills which is highly contaminated and strongly acid, succeeded by wash waters of lower acidity. Neutralisation with lime followed by vacuum filtration is recommended as the best which can be done at a reasonable cost. The lime is to be added as a 20% slurry at a rate to correspond with the flow and variation in acidity of the effluent, necessitating a flexible control of the feeding system. About 240 lb. of lime are required per 1000 gals.

C. JEPSON.

Alcohol from Nipah palm. DENNETT.—See XVIII.

PATENTS.

Fusion of aromatic sulpho-compounds. C. R. DOWNS, Assr. to WEISS & DOWNS, INC. (U.S.P. 1,658,230, 7.2.28. Appl., 30.8.24).—An alkali arylsulphonate is heated in a thin layer on a metal pan in a bath of fusible metal at 300—425° with an alkali hydroxide (2—2.5 mols. for each atom of sulphur) to yield the corresponding alkali phenoxide.

T. S. WHEELER.

Manufacture of anilides. E. A. NILL, Assr. to F. C. ACKER and J. R. DALL (U.S.P. 1,659,150, 14.2.28. Appl., 10.5.24. Cf. U.S.P. 1,659,149, p. 292).—Anilides of the higher fatty acids, e.g., stearanilide, are prepared by passage of aniline vapour through the acid at about 235°.

T. S. WHEELER.

Manufacture of derivatives of acenaphthene. I. G. FARBERIND. A.-G. (B.P. 274,902, 23.7.27. Ger., 24.7.26).—When acenaphthene-5-carboxylic or -5-sulphonic acid is nitrated, single products are obtained. 6-Nitroacenaphthene-5-carboxylic acid, m.p. 235—236°, obtained by nitration in acetic acid, is reduced by ferrous sulphate and ammonia to the amine (hydrochloride, m.p. about 300°).

C. HOLLINS.

Production of indigo-white [leuco-indigo]. J. E. SEITZ, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,657,869, 31.1.28. Appl., 25.3.24. Renewed 19.8.27).—The yield of leuco-indigo from the reduction of indigo by the action of iron and sodium hydroxide solution at 65° is improved by the addition of sodium hyposulphite or sodium sulphide near the end of the reaction.

T. S. WHEELER.

Catalytic alkylation of aromatic hydrocarbons. M. G. SHEPARD, Assr. to NAUGATUCK CHEM. Co. (U.S.P. 1,658,176, 7.2.28. Appl., 7.3.25).—See B.P. 259,507; B., 1926, 1028.

Preparation of p-hydroxy-o-benzoylbenzoic acid. NEWPORT Co. (B.P. 281,227, 15.8.27. U.S., 29.11.26).—See U.S.P. 1,654,289; B., 1928, 224.

Manufacture of diphenylguanidine. H. S. ADAMS and L. MEUSER, Assrs. to NAUGATUCK CHEM. Co. (U.S.P. 1,662,626, 13.3.28. Appl., 19.6.22).—See B.P. 199,354; B., 1924, 768.

Preparation of [mercaptobenz]thiazoles. L. B. SEBRELL and J. TEPPERA, Assrs. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,662,015, 6.3.28. Appl., 16.3.27).—See B.P. 282,947; B., 1928, 152.

Oxidation of aralkyl-substituted dithiocarbamic acids to the corresponding disulphides. W. FLEMING, Assr. to SILESIA VER. CHEM. FABR. (U.S.P. 1,662,664, 13.3.28. Appl., 16.8.27. Ger., 30.10.26).—See B.P. 279,790; B., 1928, 225.

Recovery of volatile solvents. J. H. BRÉGEAT (U.S.P. 1,663,155, 20.3.28. Appl., 1.8.25. Fr., 8.6.25).—See B.P. 251,492; B., 1926, 616.

IV.—DYESTUFFS.

Colouring matter present in the rhizomes of *Curcuma aromatica*. Salisb. B. S. RAO and V. P. SHINTRE (J.S.C.I., 1928, 47, 54 r).—The colouring matter present in the rhizomes of *C. aromatica*, Salisb., has been shown to be curcumin identical with that found in *C. longa* etc.

Light-sensitivity of dyes. II, 2. A. STEIGMANN (Kolloid-Z., 1928, 44, 249—250).—A discussion of the sensitising effect of dyes on light reactions, especially in regard to the formation of vitamins. E. S. HEDGES.

PATENTS.

Vat dyes obtained by dehalogenating polyhalogeno-violanthrones. B. MAYER and H. SIEDENBÜRGER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,658,003, 31.1.28. Appl., 28.6.26. Switz., 24.12.25).—A leuco-polyhalogeno-violanthrone or -isoviolanthrone, e.g., leuco-dichloroviolanthrone, is partially dehalogenated by treatment with alkaline reducing agents in the warm, e.g., by heating the vat to a temperature higher than is usual, to yield products which in the vat give shades similar to, but more brilliant than, those of the non-halogenated parent dyes. T. S. WHEELER.

Manufacture of modified basic dyes. J. G. KERN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,659,145, 14.2.28. Appl., 11.6.24).—A basic dye is heated in alcoholic solution in presence of mineral acid with a hydroxy-aromatic carboxylic acid, e.g., tannic acid, and the solution is poured into water to precipitate a product which dyes unmordanted wool and cotton fast shades.

T. S. WHEELER.

Manufacture of a dye powder. O. M. BISHOP and M. S. THOMPSON, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,659,131, 14.2.28. Appl., 6.6.25).—A paste of an insoluble dye is mixed with a soap, e.g., sodium stearate, a sugar, a protective colloid, and water, and after being agitated to produce a foam, the mixture is dried to yield a product which disperses in water.

T. S. WHEELER.

Manufacture of [sulphide vat] dyes. A. CATINEAU, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,662,415, 13.3.28. Appl., 19.4.27. Switz., 30.4.26).—See B.P. 270,348; B., 1927, 869.

Manufacture of vat dyes of the anthraquinone series containing the triazine ring. F. ACKERMANN, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,663,474, 20.3.28. Appl., 24.9.27. Switz., 6.10.26).—See B.P. 278,728; B., 1928, 226.

Manufacture of acylaminoflavanthrones. S. THORNLEY, and BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,662,872, 20.3.28. Appl., 3.9.27. U.K., 2.9.26).—See B.P. 282,852; B., 1928, 152.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Nitrogen content of natural and processed wools. J. BARRITT (J.S.C.I., 1928, 47, 69—72 r).—The nitrogen content of natural and processed wools has been determined, using the Kjeldahl method. For normal wools the nitrogen content varies within narrow limits, 17.07% to 16.50%, the mean value for a representative series of wools being 16.72%. The effect of a normal scouring, carbonising, or chlorination on the nitrogen content is small. Excessive treatment with alkali causes an increase in nitrogen content, sulphuric acid a decrease, and chlorination up to 10% a slight decrease. Samples of black and white wool from the same fleece (Jacob's flock) show a definite difference in nitrogen content, the black wool having the lower value (cf. Gortner, B., 1913, 939). Samples of white, brown, and black alpaca exhibit a marked gradation in nitrogen content with colour, the respective values being 17.00, 16.66, and 15.85% of nitrogen. The black alpaca after treatment with 2% sodium carbonate at 50° has a markedly increased nitrogen content, and it is suggested that the hot alkali dissolves out melanin, the melanin having a considerably lower nitrogen content than normal wool.

Microscopical investigation of artificial silk fibres. L. G. LAWRIE (J. Soc. Dyers & Col., 1928, 44, 73—78).—The preparation and mounting of fibres and cross-sections for examination, and methods of illuminating them, with avoidance of glare, are described. For the examination of the fibre entire by transmitted light and dark-ground illumination, the structural markings and appearances of different kinds of silk are classified, whilst typical interference colours shown by the different varieties under the influence of polarised light are also described. Cellulose nitrate silks show the most brilliant effects, whilst the acetate silks show no interference colours. The swelling power of artificial silks in water, as measured by the percentage increase in the diameter of the fibre, is greatest for viscose and cuprammonium silks and least for acetate silks, the fully acetylated types of the latter showing negligible swelling.

B. P. RIDGE.

Value of sisal hemp for the manufacture of marine cordage. ANON. (Bull. Imp. Inst., 1927, 25, 359—368).—Comparative tests on ropes made from Manila hemp "J" grade and first and second quality East African sisal hemp indicate that first-quality East African sisal is slightly superior and second-quality

East African sisal slightly inferior to Manila hemp "J" as regards resistance to the action of sea-water, the percentage loss in strength of sisal No. 1, Manila "J," and sisal No. 2 amounting to 63%, 69%, and 75%, respectively, after 12 months' exposure. Sisal hems, however, sink far more rapidly in water than does Manila, and show a far greater increase in girth, the percentage increase for No. 1 sisal, No. 2 sisal, and Manila "J" amounting to 9.26%, 6.21% and 3.60%, respectively, after exposure to sea-water for four months.

D. J. NORMAN.

Hemp from Cyprus. ANON. (Bull. Imp. Inst., 1927, 25, 368—370).—Cyprus hemp is only slightly inferior in chemical composition and behaviour to commercial samples of Italian hemp, its cellulose content (calculated on the dry material) being 81.5%, as against 83.8% for Italian hemp. The length of the ultimate fibre is 6—50 mm. (average 24 mm.) and its diameter 0.010—0.051 mm. (average 0.027 mm.).

D. J. NORMAN.

Lignocellulose of Victorian mountain ash (*Eucalyptus Regnans*). H. W. STRONG (J.S.C.I., 1928, 47, 87—90 r).—In the determination of the cellulose in *Eucalyptus Regnans* it is shown that it is necessary to extract the sample with alkali hydroxide, and that this is best achieved by heating at boiling temperature for not more than 1 hr. with 1% sodium hydroxide solution. More concentrated solutions of alkali give diminished yields of total cellulose with a smaller content of α -cellulose. The amount of chlorine reacting to form hydrochloric acid is approximately twice the amount of chlorine combining with the lignone, the latter being 7.5—8% of the oven-dry sample.

Bamboos from British Guiana for papermaking. ANON. (Bull. Imp. Inst., 1927, 25, 370—374).—Of three samples of bamboo from British Guiana one, consisting entirely of stems, gave, on digestion with 20% of caustic soda for 4 hrs. at 160°, 46% of pulp (on the weight of air-dry raw material) which bleached fairly readily (yield of bleached pulp 39%) and gave a paper of excellent quality and strength. This sample compared favourably with Indian *Bambusa Tulda*, and should be suitable for the commercial production of paper pulp. Of the other two samples, one gave a pulp of similar quality though in lower yield, whilst the other required more severe treatment and gave a pulp of inferior quality.

D. J. NORMAN.

Pulp and paper manufacture in North America. J. D'A. CLARK (Proc. Tech. Sect. Papermakers' Assoc., 1927, 7, 221—311).—North American pulp- and paper-making processes are described and compared with those worked in Great Britain. The advantages of using pulp in slush form are indicated, though there is no evidence that, from the point of view of strength, slush mechanical pulp is superior to baled moist mechanical pulp, or slush sulphite pulp to air-dry sulphite pulp. The relative merits of magazine grinders and pocket grinders are discussed both from the point of view of operating conditions and costs. The most satisfactory grade of pulp for newsprint is a soft well-cooked pulp made at a maximum cooking temperature of about 140°. Kraft pulp could be profitably made

in Great Britain, since a lower grade of wood can be used than is possible with the sulphite process, and higher yields of pulp are obtained. Pressures varying from 90 to 130 lb./in.² are employed, and the amount of active alkali (calculated as NaOH) may be 18–22% for kraft pulp, and about 25% for easy-bleaching sulphate pulp. The total cooking time is 4–7 hrs. with 2.5–4.5 hrs. at maximum pressure. For washing the pulp, a vacuum drum-washer, provided with a monel metal wire cloth, gives the most satisfactory results, particularly when used in conjunction with a rotary vacuum pump. Various methods which are being used for working up spent liquors are described. The monosulphite (Keebra) and semi-monosulphite (semi-Keebra) processes are in use for the treatment of wood, and their application in Great Britain for the treatment of esparto grass is suggested; a harder and stronger pulp is obtained in considerably higher yields than is given by the soda process. It is impractical to use the monosulphite process alone, owing to the unwieldy accumulation of thiosulphates in the liquor during recovery and regeneration. When, however, both processes are used, this difficulty can be avoided to a large extent by using as much as possible of the black liquor from the full monosulphite cook as a diluent for the cooking liquors for both full and semi-monosulphite cooks; further, in the recovery of the semi-monosulphite black liquor, the ash should be burnt without excessive firing in order to avoid the formation of sulphides (and hence of thiosulphates during the sulphiting operation), and the sulphite liquor should, as far as is practicable, be prepared from fresh caustic soda. Methods of testing mechanical and chemical pulps for strength are given, and modern tendencies in the design of pulp- and paper-making machinery are outlined. D. J. NORMAN.

Vanillin from sulphite-cellulose waste liquors. KÜRSCHNER.—See III.

PATENTS.

Recovery of waste fibrous material. L. KIRSCHBRAUN (U.S.P. 1,659,401, 14.2.28. Appl., 16.8.22. Renewed 30.6.26).—Waste fibrous stock containing bitumen, e.g., waste from the process described in B.P. 174,114 (B., 1922, 213 A), is agitated with bentonite and water at 95° to remove bitumen. T. S. WHEELER.

Production of cellulose by decomposition of vegetable fibres. O. C. STRECKER (U.S.P. 1,658,213, 7.2.28. Appl., 21.9.25. Ger., 30.9.24).—The fibres are heated at about 100° with a solution of an alkali salt of a homocyclic hydroxy-compound, e.g., sodium phenoxide. T. S. WHEELER.

Manufacture of cellulose compounds [viscose silk]. F. C. NIEDERHAUSER and H. B. KLINE (U.S.P. 1,655,626, 10.1.28. Appl., 21.12.26).—The addition of a small proportion (0.2%) of an alkali salt of a sulphated vegetable or animal oil to viscose solution lowers the surface tension and thus facilitates spinning. T. S. WHEELER.

Manufacture of viscose. J. M. LEAVER, Assr. to PACIFIC LUMBER Co. (U.S.P. 1,659,033, 14.2.28. Appl., 23.12.24).—The live bark of the *Sequoia* is used as a source of cellulose for artificial silk and film production. H. ROYAL-DAWSON.

Recovery of poisonous gases [hydrogen sulphide] in viscose manufacture. F. C. NIEDERHAUSER and H. B. KLINE, Assrs. to INDUSTRIAL FIBRE Co. (U.S.P. 1,655,709, 10.1.28. Appl., 9.3.27).—Evolution of hydrogen sulphide from viscose solution during spinning is inhibited by treating the concentrated solution prior to dilution and precipitation with carbon dioxide. The hydrogen sulphide and carbon disulphide evolved in the ensuing decomposition of sodium trithiocarbonate are absorbed in alkali solution. T. S. WHEELER.

Improvement of artificial fibres consisting of regenerated cellulose. HEBERLEIN & Co. A.-G. (B.P. 264,529, 15.1.27. Ger., 15.1.26).—The lustre and other properties of cellulose artificial silk are modified by treating the silk in fibre or fabric form with mercerising solutions (other than caustic alkali solution of or above 18% strength) in the absence of sulphonic acids of the benzene or naphthalene series, but optionally in the presence of restrainers such as mono- and poly-hydric alcohols, heterocyclic bases, etc. Suitable mercerising solutions are alkali hydroxide solution above d 1.075 and below d 1.21, sulphuric acid of $d > 1.41$, phosphoric acid of $d > 1.53$, hydrochloric acid of $d > 1.162$, nitric acid of $d > 1.32$, mixtures of these acids, and concentrated salt solutions. With caustic soda solution of d 1.162 at the ordinary temperature 4 sec. is a suitable time of treatment. The treated fabric shows not only a modified lustre, but also increased stiffness and a greater uniformity of structure. Mixtures of nitric and sulphuric acids containing less than 20% of water produce a non-translucent lustreless effect when nitric acid predominates, and a soft, broken-up, lustrous effect, resembling crêpe, when sulphuric acid predominates. D. J. NORMAN.

Manufacture of products having a basis of cellulose derivatives. W. A. DICKIE and J. H. ROONEY, Assrs. to CELANESE CORP. OF AMERICA (U.S.P. 1,655,677, 10.1.28. Appl., 21.11.25. U.K., 10.12.24).—The process of B.P. 249,946 (B., 1926, 532) is modified in that the plasticising of a coloured cellulose derivative, e.g., cellulose acetate, is performed in presence of a moulding powder comprising, e.g., cellulose acetate and monomethylxylenesulphonamide. T. S. WHEELER.

Cellulose ether composition. S. J. CARROLL, Assr. to EASTMAN KODAK Co. (U.S.P. 1,658,369, 7.2.28. Appl., 7.10.25).—A flexible, transparent, waterproof sheet of value in the manufacture of photographic film is formed of an alkylcellulose, e.g., ethylcellulose (100 pts.), and a glyceryl acetate, e.g., triacetin (5–50 pts.). T. S. WHEELER.

Manufacture of articles of plastic material. A. LANDUCCI, Assr. to DU PONT-PATHÉ FILM MANUF. CORP. (U.S.P. 1,658,725, 7.2.28. Appl., 8.2.26. Fr., 20.2.25).—A cellulose ester or ether, e.g., cellulose acetate, dissolved in an organic solvent miscible with water, e.g., acetone, is precipitated in an aqueous bath, and the opaque product is rendered transparent by treatment with acetone or similar solvent. T. S. WHEELER.

Rejuvenation of cellulose ester plastic. J. H. SHERTS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,657,870, 31.1.28. Appl., 1.11.23. Renewed 1.8.27).—Brittle nitrocellulose plastic is rendered flexible

by immersion for about 1 min. in a mixture of a high-boiling solvent, *e.g.*, amyl acetate, and a low-boiling solvent, *e.g.*, benzene. The treated material is dried at about 38°. T. S. WHEELER.

Reducing the viscosity characteristics of cellulose acetate. L. E. BRANCHEN and C. U. PRACHEL, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,658,368, 7.2.28. Appl., 6.10.26).—Cellulose acetate dissolved in formic or acetic acid is subjected at room temperature for about 50 hrs. to the action of ultra-violet light.

T. S. WHEELER.

Apparatus for the manufacture of artificial silk. F. W. SCHUBERT, Assr. to BRYSSILKA, LTD. (U.S.P. 1,662,478, 13.3.28. Appl., 7.7.27. U.K., 2.7.25).—See B.P. 258,371; B., 1926, 1009.

Manufacture of compositions with cellulose derivatives. H. DREYFUS (U.S.P. 1,661,169, 6.3.28. Appl., 26.5.23. U.K., 13.7.22).—See B.P. 205,195; B., 1923, 1172 A.

Machine for washing wool and other textile materials. G. HEINTZE (B.P. 271,063, 2.5.27. Ger., 15.5.26).

Nozzles for use in the production of artificial [silk] filaments. C. L. WALKER (B.P. 286,342, 19.11.26. Addn. to B.P. 253,209; B., 1926, 661).

Washing apparatus for use with parchmentising and like machines. P. ERKENS (B.P. 286,506, 9.5.27).

Treatment of colloids (B.P. 283,686).—See XIV. **Catgut substitute** (B.P. 285,752).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Treatment [weighting] of artificial silks. R. CLAVEL (B.P. 280,094, 30.3.27. Addn. to B.P. 277,602; B., 1928, 154).—Artificial silk (especially acetate silk) is given an acid reaction by immersion in an acid solution (phosphoric acid, chloroacetic acid, aluminium sulphate, etc.) with or without addition of glue or other protective colloid. The intermediate washing between impregnation and precipitation is thus rendered unnecessary, no precipitation occurring on the surface of material so treated. Alternatively, acid and/or protective colloid may be added to the impregnating bath or to the fibre before precipitation. The acid may be subsequently removed by means of a mild alkaline bath, to which hæmatein, or other dyes containing tannin, may be added. C. HOLLINS.

Dyeing process [for fibres]. A. ESCHACH and P. J. WORMS (U.S.P. 1,661,606, 6.3.28. Appl., 20.11.23. Ger., 25.11.22).—See B.P. 227,906; B., 1925, 204.

Increasing the affinity of animal fibres for dyestuffs. E. STOCKER, Assr. to CHEM. WORKS, FORMERLY SANDOZ (U.S.P. 1,662,404, 13.3.28. Appl., 27.10.27. Ger., 20.11.26).—See B.P. 245,759; B., 1927, 475.

Dyeing [cotton] with basic dyes involving the use of arylamides of aromatic hydroxycarboxylic acids as mordants. A. GUENTHER, J. HALLER, and E. KÖSTER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,663,451, 20.3.28. Appl., 29.1.26. Ger., 19.2.25).—See G.P. 441,326; B., 1927, 905.

Dyeing of cellulose esters or ethers. R. S. HORSFALL, L. G. LAWRIE, and J. HILL, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,662,514, 13.3.28. Appl., 2.3.27. U.K., 11.6.26).—See B.P. 275,373; B., 1927, 776.

Metallising process [for textiles]. J. VON BOSSE (U.S.P. 1,661,517, 6.3.28. Appl., 6.5.26. Ger., 27.5.25).—See B.P. 252,710; B., 1927, 820.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Alteration of concentrated solutions of sodium bisulphite. E. ISNARD (J. Pharm. Chim., 1927, [viii], 6, 211—212).—A concentrated solution of sodium bisulphite, prepared in 1923, when added to an aqueous sodium thiosulphate solution produced a precipitate and hydrogen sulphide was evolved. The bisulphite solution, which originally contained 38% Na₂SO₃, was found to have the following composition: water 60.7%, Na₂SO₃ 1.27%, NaHSO₄ 16.54%, Na₂SO₄ and sulphur (traces) 11.49%. E. H. SHARPLES.

Manufacture of ammonium chloride from ammonium sulphate and common salt. E. HOCHBERGER (Chem.-Ztg., 1928, 52, 22—23).—Common salt is added to a saturated solution of ammonium sulphate at the boiling point. Sodium sulphate separates, and is washed with a solution of sodium sulphate saturated at 32.4° to remove adherent liquor and ammonium chloride. The mother-liquor is cooled to 40°. Ammonium chloride separates and is recrystallised. The liquors are used repeatedly in cycle. S. I. LEVY.

Decomposition of alkaline-earth sulphates. L. E. BHATT and H. E. WATSON (J. Indian Inst. Sci., 1927, 10A, 117—129).—When calcium sulphate is heated with carbon, the sulphide formed begins to react with the sulphate at about 950° with evolution of sulphur dioxide, but even at 1150° this reaction is relatively slow. The amount of sulphur as dioxide and trioxide which is expelled from calcium sulphate by heating with silica increases with increase in the relative amount of the latter, and with 2 mols. of silica per mol. of sulphate decomposition is nearly complete in 4 hrs. at 1100°. Regrinding the reacting mixture after it has been heated for some time much facilitates reaction, whereas moisture has an unfavourable effect. Addition of carbon to the mixture of silica and sulphate results in a decrease in the amount of sulphate decomposed, probably because of the formation of sulphide, which reacts with silica less readily than does sulphate. At the same time, however, there is an increase in the quantity of volatile sulphur compounds formed, possibly owing to the formation of an intermediate product, perhaps the sulphite, which is more reactive towards silica than is the sulphate. The presence of iron in a reacting mixture of silica and calcium sulphate is not advantageous on account of the somewhat extensive sulphide formation, and it appears that the maximum amount of volatile sulphur compounds is secured by heating with silica only. Calcium sulphite at 950° decomposes chiefly according to the equation $4\text{CaSO}_3 = 3\text{CaSO}_4 + \text{CaS}$; silica almost completely prevents the formation of sulphide. The decomposition points of the sulphates of magnesium, strontium, and

barium when heated in air are at 740°, 1100°, and above 1100°, respectively. Silica promotes the decomposition of magnesium sulphate to a considerable extent, but with barium sulphate the effect is much less, and with strontium sulphate least of all. R. CUTHILL.

Production of ammonium phosphates from secondary and tertiary calcium phosphates. C. MÜCKENBERGER (Z. anorg. Chem., 1928, 169, 81—95).—Tricalcium phosphate readily reacts at 180—220° and under 11—17 atm. pressure with phosphoric acid, only a slight excess of acid being required, but equilibrium is established when a large part of the phosphate has been converted only into dicalcium phosphate. In presence of ammonium sulphate, however, the yield of soluble phosphate is increased, owing to the reaction $\text{Ca}_3(\text{PO}_4)_2 + 3(\text{NH}_4)_2\text{SO}_4 + 4\text{H}_3\text{PO}_4 = 6\text{NH}_4\text{H}_2\text{PO}_4 + 3\text{CaSO}_4$, and with an amount of phosphoric acid about 40% more than the theoretical amount the whole of the phosphate is rendered soluble. It is suggested that a valuable artificial fertiliser might be manufactured from natural phosphate by opening it up in this way, then converting the calcium sulphate into ammonium sulphate by one of the available methods, and using part of the filtrate, after it has been heated so as to drive off some ammonia and give a strongly acid product, to open up fresh phosphate. For this purpose a considerable excess of monoammonium phosphate is required, but the temperature need not exceed 100°; diammonium phosphate has very little solvent action on tricalcium phosphate. The action of ammonia and carbon dioxide on dicalcium phosphate apparently involves a number of simultaneous reactions, an equilibrium finally being set up between an ammoniacal solution of ammonium phosphate and unchanged dicalcium phosphate, tricalcium phosphate, calcium carbonate, and a little calcium hydroxide. Under the most favourable conditions a yield of 50% of soluble phosphates is obtained. R. CUTHILL.

Arsenious oxide in solution. MILLAR.—See XX.
Oxidation of ammonia to nitric acid. FOWLER and others.—See XXIII.

PATENTS.

[Preparation of silica gel for] the recovery of gas and vapours from gas mixtures. C. F. BOEHRINGER & SÖHNE G.M.B.H., and E. WILKE (Swiss P. 119,220—1, 20.10.25. Ger., 3.11.24).—An adsorbent silica gel is obtained by treating a solution of an alkali silicate with (A) a solution of a salt of a metal the silicate of which is only slightly soluble in water, or (B) with carbon dioxide. In either case the gelatinous precipitate is collected, washed, dried, and dehydrated at 200—500°.

A. R. POWELL.

Collection of phosphoric acid. B. G. KLUGH, Assr. to FEDERAL PHOSPHORUS Co. (U.S.P. 1,659,146, 14.2.28. Appl., 7.12.20. Renewed 12.5.27).—The gases containing phosphoric acid obtained by processes such as those of U.S.P. 1,492,713 and 1,497,727 (B., 1924, 557,632) are freed from phosphoric acid by passage through a filter-bed of sand, from which phosphoric acid is periodically removed by treatment with a current of air or water. The dilute solution obtained by the

latter process can be concentrated by treatment with the hot gases passing to the filters. T. S. WHEELER.

Manufacture of anhydrous magnesium chloride. R. GRIESSBACH and K. RÖHRE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,661,894, 6.3.28. Appl., 22.11.26. Ger., 7.12.25).—Crystals of the chloride are treated in a gaseous mixture containing 10—25% HCl at 200—350°.

H. ROYAL-DAWSON.

Manufacture of zirconium compounds. C. J. KINZIE, Assr. to TITANIUM ALLOY MANUF. Co. (U.S.P. 1,658,807, 14.2.28. Appl., 27.5.26).—A siliceous zirconium ore is fused with potassium hydroxide at 950°, and the product is treated with dilute sulphuric acid at 65° to form a solution from which, on cooling, potassium zirconium sulphate separates. Alternatively, the ore is fused with sodium carbonate, and potassium chloride is added after treatment with sulphuric acid.

T. S. WHEELER.

Recovery of sulphur. I. G. FARBENIND. A.-G. (Swiss P. 119,973, 7.11.25. Ger., 6.7.25).—The solution of sulphur in a suitable solvent is allowed to fall through a heated vessel on to a plate heated at such a temperature that the solvent is distilled away and the separated sulphur is melted.

A. R. POWELL.

Catalytic oxidation of sulphur dioxide. A. O. JAEGER and J. A. BERTSCH, Assrs. to SELDEN Co. (U.S.P. 1,657,753—4, 31.1.28. Appl., [A] 6.2.26, [B] 15.2.26).—(A) A calcium, ferric, or other zeolite is treated with an alkali vanadate, tungstate, or chromate solution at 60° to give a product of value as a catalyst for the oxidation of sulphur dioxide. (B) Potassium vanadate solution is neutralised with dilute acid and treated with sodium silicate and hydrochloric acid at 70°, and the product is treated with copper sulphate, calcium chloride, or ferric sulphate solution.

T. S. WHEELER.

Extraction of phosphorus from its compounds by reduction. H. WITTEK (B.P. 275,145, 20.11.26. Ger., 27.7.26).—Phosphorus is produced concurrently with calcium cyanamide by mixing a phosphorus compound, other than a heavy-metal compound, with calcium or other carbide, and heating in a furnace in a current of nitrogen, the heat of formation of the cyanamide being utilised for the reduction. W. G. CAREY.

Production of water-gas and hydrogen. SYNTHETIC AMMONIA & NITRATES, LTD., and H. A. HUMPHREY (B.P. 282,573, 22.2.27).—Semi-coke in lump form is made from slack coal of high caking power by carbonising below 600° after preheating in presence of oxygen to control its caking properties. The semi-coke, which may be further heated at 800—900° to drive off volatile matter, is fed direct from the retort to a water-gas generator operated with excess of steam and having a shallow fuel bed, and the resulting gas mixture is compressed and treated with excess of steam above 800° in the presence of a catalyst, e.g., nickel, to convert methane into carbon monoxide and hydrogen. If hydrogen for ammonia synthesis is desired the carbon monoxide is converted into carbon dioxide by treatment with steam in the presence of an iron oxide catalyst at about 500°, the carbon dioxide being removed, preferably, by dissolution in water under pressure.

W. G. CAREY.

Manufacture of hydrogen peroxide. H. S. TAYLOR, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,659,382, 14.2.28. Appl., 1.2.26).—An equimolecular mixture of hydrogen and oxygen containing mercury vapour is passed through a quartz tube, in which it is subjected to the action of the rays emitted by a mercury vapour lamp. T. S. WHEELER.

Production of argon. W. DANNENBAUM, Assr. to PACIFIC NITROGEN CORP. (U.S.P. 1,658,631, 7.2.28. Appl., 13.10.23).—A mixture of argon and nitrogen is passed with excess of hydrogen under suitable conditions of temperature and pressure over a catalyst adapted to induce formation of ammonia, and the mixture of hydrogen and argon resulting is treated with excess of oxygen to form water, the argon being finally purified from oxygen by treatment with copper.

T. S. WHEELER.

Rapid evaporation to dryness of ammonium nitrate solutions. C. TONIOLO (U.S.P. 1,661,203, 6.3.28. Appl., 27.1.26. It., 7.2.25).—See B.P. 247,228; B., 1927, 408.

Purifying and bleaching of heavy spar. K. EBERS (U.S.P. 1,663,159, 20.3.28. Appl., 22.12.25. Ger., 24.12.21).—See B.P. 245,155; B., 1926, 742.

Manufacture of aluminium compounds. G. MUTH (U.S.P. 1,661,618, 6.3.23. Appl., 29.3.21. Ger., 13.5.19).—See G.P. 319,420; B., 1920, 517 A.

Hydrogen-ion comparator (U.S.P. 1,659,529).—See I. **Preparation of hydrogen** (F.P. 621,902).—See II. **Electrolysis of water** (Swiss P. 120,249). **Electrolysis of salt solutions** (G.P. 446,009).—See XI. **Base-exchange substances** (G.P. 445,377—8).—See XXIII.

VIII.—GLASS; CERAMICS.

Ceramic colours and their use in vitreous enamels. W. N. HARRISON and T. O. HARTSHORN (J. Amer. Ceram. Soc., 1927, 10, 747—760).—The production of many colouring stains, including such substances as cobalt oxides, chromic oxides, cadmium sulphide and selenide, and copper oxide as bases, and their incorporation in vitreous enamel bodies, are discussed. It is stated that removal of soluble matter from the calcined stains, especially selenium-red stains, by washing prevents a spotted appearance in the fired enamels. It was found necessary to duplicate the time and temperature of calcination of the stains, and the furnace atmosphere in order to duplicate the colour obtained with a given batch composition; to such variations, again, selenium-red stains are very sensitive. Opacifiers introduced either in the raw batch or mill batch generally tend to lighten the colour produced by a given stain. Some materials, however, probably due to chemical reaction have a counteracting tendency to darken the enamel. Cryolite and felspar in the frit have little effect on the shades obtained except a slight lightening of tone. On the other hand, sodium antimonate produces a darker tone in spite of its tendency to opacity; this effect is accentuated by the substitution of silica for felspar. Lead oxide darkens the shade of stains, especially red stains. A. T. GREEN.

Uranium oxide colours and crystals in low-temperature glaze combinations. J. R. LORAH (J. Amer. Ceram. Soc., 1927, 10, 813—820).—Glazes containing lead, sodium, and boron combined with other inorganic compounds were compounded according to recipe and fired to a definite maturing temperature in an oxidising atmosphere. Colours varying from yellow to black were obtained. It is shown that a small quantity of uranium oxide in a lead glaze produces a yellow colour, 5% a bright orange, and 10% a dark orange colour. Black spots, noted in certain glazes, are due to the reduction of the yellow uranium oxide to the black oxide. Glazes containing, additionally, boron compounds with silica give yellow glazes with a greenish tinge and show no tendency towards the formation of orange colours or crystalline structures. Lead oxide, owing to its volatility during firing, appears to be essential to the production of crystals. Two types of crystals depending on the ratio of the uranium to the lead content are noted. The effect of adding such substances as clay, calcium fluoride and carbonate, and barium carbonate appears to be to decrease the amount of lead which must be volatilised before crystal formation commences. A. T. GREEN.

Dehydration and firing behaviour of clays. R. F. GELLER and W. H. WADLEIGH (J. Amer. Ceram. Soc., 1927, 10, 929—955).—Results of laboratory studies of the drying and water-smoking behaviour of twelve clays used in the manufacture of bricks and tiles are correlated with data obtained on works where the clays are in actual use. As the result of preliminary drying tests, two equations were developed by means of which the air temperature and the relative humidity could be calculated for any given schedule. In the drying tests the maximum temperature reached was 110° and the minimum relative humidity 4%. The water-smoking tests were carried out in an electrically-heated furnace specially constructed to enable the amount of loss in weight and the temperature lag within the specimens to be observed at intervals of 15 min. In the works' tests thirteen kilns were investigated; two tests were made, one under conditions of normal practice and the other with kiln setting and firing method modified in accordance with conclusions based on the results of the first firing. The shortest time in which the drying shrinkage could be completed without injury to the specimens varied from 4 to 42 hrs.; the fire clays were much more difficult to dry than the shales and surface clays. No direct relation was found between the elimination of shrinkage and pore water and the removal of hygroscopic and chemically combined water. It is concluded that clay wares such as bricks and paving blocks could be heated with safety to 1000° if proper precautions were taken, but in practice a longer period is necessary owing to the setting of insufficiently dried ware, the limitations of kiln construction and design, and the impracticability of removing promptly all water vapour from the kiln. F. SALT.

Cyanite and diasporite refractories. E. J. VACHUSKA and G. A. BOLE (J. Amer. Ceram. Soc., 1927, 10, 761—773).—Mixtures containing diasporite-cyanite, di-

aspore-clay, and cyanite-clay were made, and methods for manufacturing and testing such refractories are examined. Diaspore products show a permanent shrinkage after prolonged subjection to high temperatures. Such after-shrinkage, probably due to an inversion of the diaspore to corundum, and accompanied by an increase in specific gravity (3.3—4.0), can be prevented either by the addition of 20% or more cyanite to the diaspore mixtures, or the precalcination of the diaspore to as high a temperature as possible. The addition of cyanite, however, must be carefully controlled so that the resistance to thermal shock is not impaired. Data concerning the linear shrinkage, porosity, and modulus of rupture of the fired products are given.

A. T. GREEN.

Spalling and loss in compressive strength of firebrick. H. R. GOODRICH (J. Amer. Ceram. Soc., 1927, 10, 784—794).—The loss in compressive strength of a brick, which is the result of thermal shock attending repeated heat-treatments at high temperatures (1250° and 1350°) and coolings in air, is suggested as a satisfactory criterion of spalling tendency. It is indicated that the development of a vitrified structure markedly predisposes the material to spalling. Whilst a material with a high quartz content and a clay bond possesses a comparatively low initial strength, yet it retains a higher percentage of strength after thermal shock than do the fireclay products investigated. Pre-treatment at high temperatures, by inducing extra vitrification, helps the failure of certain fireclay products in the spalling test. A review of methods of testing spalling tendency is given.

A. T. GREEN.

Electrical resistance of porous materials. KNOWLER.—See XI.

PATENTS.

Manufacture of refractory products. W. A. FARISH, ASSR. to BUFFALO REFRACTORY CORP. (U.S.P. 1,658,406, 7.2.28. Appl., 31.8.22).—A mixture of graphite and an electric furnace refractory product, e.g., silicon carbide, is impregnated with a carbonaceous binder, formed, carbonised, and dipped in a liquid flux, e.g., sodium silicate.

T. S. WHEELER.

Manufacture of glass in continuous sheets by flowing and rolling. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST. GOBAIN, CHAUNY, & CIREY (B.P. 280,582, 11.11.27. Fr., 15.11.26).

IX.—BUILDING MATERIALS.

Composition of alite. O. REBUFFAT (Giorn. Chim. Ind. Appl., 1927, 9, 520).—Objection is raised to the formula $8\text{CaO} \cdot \text{SiO}_2 \cdot \text{Al}_2\text{O}_3$, proposed by Jänecke for alite, the principal constituent of anhydrous Portland cement (B., 1926, 747), mainly on the ground of the difference in composition between ordinary Portland cement and that corresponding with the above formula.

T. H. POPE.

Electrical resistance of porous materials. KNOWLER.—See XI. **Concrete for water reservoirs.** MABEE.—See XXIII.

PATENTS.

Manufacture of aluminous cement. E. C. R. MARKS. From G. POLYSIUS (B.P. 286,122, 6.5.27).—

The raw unground materials are fed into a rotary retort, the size of the pieces used being dependent on their physical and chemical nature. H. ROYAL-DAWSON.

Preservation of wood and solutions therefor.

L. P. CURTIN, ASSR. to CURTIN-HOWE CORP. (U.S.P. 1,659,135, 14.2.28. Appl., 15.5.26).—A mixture of zinc chloride, calcium acetate, arsenic trioxide, and dilute acetic acid is of value as an insecticide and fungicide; zinc meta-arsenite is deposited on exposure to air.

T. S. WHEELER.

Production of asphalt-paving mixtures. J. S. DOWNWARD (U.S.P. 1,662,377, 13.3.28. Appl., 11.8.23).—Separately prepared mixtures of mineral matter with blown asphalt of high and low m.p., respectively, are mixed while cold, and the mixture is heated at the place of use, whereby a mixture of standard asphalt content is produced.

J. S. G. THOMAS.

Road binder. M. F. COUGHLIN (U.S.P. 1,662,299, 13.3.28. Appl., 24.2.22).—The binder consists of a basic bituminous material, sulphite liquor, and lactic acid.

H. ROYAL-DAWSON.

Production of porous [heat- and sound-]insulating materials. E. C. BAYER (B.P. 265,968, 8.2.27. Denm., 9.2.26).—A foam of a solution of organic matter, e.g., glue, chrome-gelatin, caseinates, etc., is produced by means of soap, saponin, or similar material, or by adding to the organic matter foam previously prepared from such substances; in the latter case the strength of the foam is increased by adding gelatin or glycerol.

W. G. CAREY.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Chemical composition and roasting of a Siegerland spathic iron ore. W. STAHL (Chem.-Ztg., 1928, 52, 162).—In the ordinary roasting process iron pyrites is mainly oxidised, but the copper sulphide sometimes present is difficult to decompose and passes on to the blast furnace, where its presence is objectionable. Chloride and sulphide roasting, with subsequent leaching to remove copper, are suggested.

C. J. SMITHELLS.

Air-hardening of rivet steels. H. K. HERSCHEMAN (U.S. Bur. Stand. Tech. Paper No. 358, 1927, 22, 141—169).—The suitability of air-hardening nickel-chromium sheets for riveting armour plate has been examined, and the effect of varying the carbon content and of the addition of manganese and molybdenum on the ballistic resistance has been determined. The values obtained in the shear test are a good indication of the ballistic and impact properties of a steel for use as rivets. The most satisfactory results are obtained with steels containing 4—6% (Ni + Cr) provided that the carbon content is within the limits 0.17—0.25%. For the steel with 3.5% Ni and 1.5% Cr the optimum impact resistance is obtained with 0.20% C. Equally good results at a slightly lower cost may be obtained with smaller quantities of nickel and chromium provided that a small proportion of molybdenum or manganese or both is added, the carbon content also being suitably adjusted. With too high a carbon content there is a tendency for long-shanked rivets to become loose owing

to the expansion caused by the slow change from austenite to martensite which takes place at relatively low temperatures in these steels.

A. R. POWELL.

Contraction of steel castings. F. KÖRBER and G. SCHITZKOWSKI (*Stahl u. Eisen*, 1928, **48**, 129—135, 172—178).—In making steel castings a total shrinkage of about 2% must be allowed for; even with this allowance sound castings of complicated design are not obtained unless certain conditions are observed. During the cooling of a casting with flanges or other projecting parts, fracture usually occurs almost immediately after the casting has solidified, *i.e.*, between the f.p. of the metal and 800°. Steel with the lowest possible content of sulphur and phosphorus should be used, as these elements usually segregate to the weakest parts of the castings and almost invariably cause fracture; addition of 0.6—0.8% Mn to the alloy almost entirely removes any trouble likely to be caused by sulphide segregation.

A. R. POWELL.

Yield point in steel at various temperatures. J. MUIR (*J. Roy. Tech. Coll., Glasgow*, 1927, [4], 14—23).—The yield points at various temperatures have been determined for mild steel in the form of wire. The temperatures required were obtained by electrically heating the wires. The rate of extension at a yield point is very greatly increased by increase of temperature, whilst the phenomenon of the yield point (*i.e.*, the extension of the wire by jerks and not by creeping) disappears entirely at 250°, in opposition to the observations of Lea and Crowther (*Engineering*, 1914, **98**, 487).

L. M. CLARK.

Determination of the permanent strength of steel at high temperatures. F. KÖRBER (*Z. Metallk.*, 1928, **20**, 45—49).—The permanent strength of a metal at high temperatures is defined as the load per unit area that will just continue to produce an elongation when constantly applied; this value largely determines the suitability of a constructional material to withstand constant high pressures at elevated temperatures. For practical purposes the load which causes an increase of not more than 0.001% in the elongation in the period between the third and sixth hour after application is determined. To carry out this work the rate of increase of elongation is determined for a number of different loads and the results are plotted against the load. A machine and furnace suitable for the determination of small increases in the length of a specimen under constant load at high temperatures are illustrated, and examples are given of the results obtained with copper, zinc, and several steels.

A. R. POWELL.

Stepped lowering of the A1 transformation in steels. T. MURAKAMI (*Sexagint [Osaka celebration]*, Kyoto, 1927, 171—179).—The appearance of the A1 transition in steel at definite temperatures governed by the rate of cooling and the maximum temperature of heating is explained on the basis of Tammann's theory of the velocity of crystal growth.

H. F. GILLBE.

Density of hot-rolled and heat-treated carbon steels. H. C. CROSS and E. E. HILL (*U.S. Bur. Standards Sci. Paper No. 562*, 1927, **22**, 451—466).—The density of commercially pure and electrolytic iron,

and of carbon steels containing 0.09—1.29% C, was determined after hot-rolling, annealing, quenching, and tempering. The density of pure iron was 7.864, and of hot-rolled carbon steels containing up to 1.3% C was 7.855—0.032C, where C is the carbon content. A similar decrease in density with increasing carbon content was found for annealed steels, for which the value of *d* was 7.860—0.04C. Quenching decreases the density owing to the formation of martensite, and is most marked in the 1% carbon steel. The density increases on tempering and approaches that of the annealed material when tempered above 600°.

C. J. SMITHELLS.

Practical problems of corrosion. III. Formation of rust and its consequences. IV. Corrosion of wrought iron in relation to that of steel. V. Corrosion and protection at the contacts of dissimilar metals. U. R. EVANS (*J.S.C.I.*, 1928, **47**, 55—62 T, 62—69 T, 73—77 T).—III. Rust is not the direct corrosion product of iron, but a precipitate, mainly consisting of iron hydroxides; it is usually formed at a sensible distance from the site of corrosion and varies in character with the conditions of precipitation. Hard waters produce calcium and magnesium compounds on the cathodic portions of rusting specimens, and these substances obstruct the access of oxygen to the metal, thus reducing the rate of corrosion; the corrosion of steel half immersed in magnesium sulphate solution of various concentrations has been measured. The "apparent volume" of the "rust sludge" produced by numerous liquids has also been determined; it may be several hundred times the volume of the iron destroyed, thus explaining the rapid choking of pipes. The true volume of rust also exceeds that of the iron destroyed, and the expansion may sometimes cause disintegration where the rust is precipitated within the metal, or in crannies between plates. This is more likely to occur in atmospheric attack than in immersed corrosion, but, if a sufficient margin of strength has been provided, the expansion will more often tend to plug the pore or crevice with rust, thus putting an end to the action.

IV. When deeply immersed in acids, wrought iron is attacked, as a whole, quicker than steel, but the corrosion proceeds most quickly when it reaches the internal portions; the material consists of alternate resistant and corrodible layers, the latter containing obstructive flaky particles. The action of drops of dilute sulphuric acid (such as drops of rain in coal-burning districts) is much the same on wrought iron as on steel. Specimens of wrought iron, half immersed in solutions of salts, are attacked rather more slowly than specimens of steel, but the difference is small. On the other hand, wrought iron appears to have a definite advantage for buried work (*e.g.*, pipes) which has to withstand anodic attack from stray electric currents in the presence of chlorides; the total amount of metal destroyed at places where discontinuities occur in the protective tar or other covering is determined by Faraday's law, and under similar conditions will be the same for both materials; but whereas the attack on steel burrows downwards, producing smooth, rounded grooves, the attack on wrought iron will usually tend to be diverted sideways by the obstructive layers, and thus perforation is likely—

under ordinary circumstances—to be deferred for a longer time if wrought iron is employed.

V. A series of potential measurements has been made between steel and seven other metals in different liquids in presence of oxygen. Zinc and cadmium are usually anodic towards steel; copper and nickel are usually cathodic, whilst tin, lead, and aluminium are variable. Measurements of the enhancement of the corrosion of steel by contact with copper, nickel, and lead plates of different sizes indicate that copper is the most dangerous and lead the least dangerous. Contact with zinc was found to afford complete protection to steel in most of the liquids tested, but the amount of zinc consumed—although low in liquids containing calcium salts—was found, in solutions of sodium chloride or sulphate, to be actually greater than the consumption of steel which took place when zinc was not present.

Disposal of acid iron wastes from a steel mill. S. E. COBURN (*Ind. Eng. Chem.*, 1928, 20, 248–249).—These wastes consisted of spent acid liquor from pickling steel, rinse waters from washing the metal after pickling, and spent lime solution from neutralising any acid adhering to the metal after rinsing. The rinse water and lime solution could be passed into the sewer after treatment, but in the case of the spent acid it is recommended to concentrate it and, after recovering ferrous sulphate, either return the mother-liquor to the pickling vat or neutralise the free acid with scrap iron and recover the ferrous sulphate so produced. C. JEPSON.

Gravimetric determination of titanium in iron alloys. Separation [of titanium] from iron. J. CIOCHINA (*Z. anal. Chem.*, 1928, 73, 40–46).—The alloy (0.5–10 g.) is dissolved in aqua regia and the solution evaporated with 5–20 c.c. of sulphuric acid (*d* 1.8) until copious fumes are evolved. The solution is cooled, diluted with 100 c.c. of cold water, neutralised with sodium carbonate, treated with 2 c.c. of sulphuric acid (1:1) and 6 g. of sodium thiosulphate, and poured slowly into 400 c.c. of boiling water. After boiling for 1–2 min. the liquid is cooled, filter-pulp added, and the precipitate collected, washed with hot water, and ignited to titania. The use of an excess of thio-sulphate is stated to prevent precipitation of part of the titania. A. R. POWELL.

[Iron] alloys with a high content of nickel and chromium. P. CHEVENARD (*Rev. Mét.*, 1928, 25, 14–34).—A review of the present state of our knowledge of the thermal, electrical, and mechanical properties of nickel-chromium and iron-nickel-chromium alloys, and a brief account of the valuable properties of commercial alloys in these groups. A. R. POWELL.

Anomalies in nickel-steels and their applications. C. E. GUILLAUME (*Rev. Mét.*, 1928, 25, 35–43).—A review of the author's work on iron-nickel alloys during the past 30 years, with an account of the practical application of the peculiar thermal and magnetic properties of these alloys to industrial uses. A. R. POWELL.

Rapid determination of silica, alumina, lime, and magnesia in low-manganese iron ores. R. P. HUDSON (*Chemist-Analyst*, 1927, 16, [2], 4–8).—Aluminium is precipitated as phosphate with sodium ammonium phosphate and sodium thiosulphate; lime

and magnesia are determined in an aliquot part of the filtrate. The magnesium ammonium phosphate is corrected for manganese. CHEMICAL ABSTRACTS.

Detection of cobalt in steel. J. MOIR (*J. S. African Chem. Inst.*, 1928, 11, 33–34).—The steel (10 mg.) is dissolved in 8 c.c. of aqua regia and the solution examined with a direct-vision spectroscope, using a wide slit; with 5% Co or more the red end of the spectrum is cut off entirely and two narrow absorption bands (λ 6210 and 6070) are visible in the orange. The latter are not seen with less than 5% Co, but two deep bands appear in the red at 7000 and 6610. Addition of water destroys the characteristic absorption effect of cobalt.

A. R. POWELL.

Anomaly of reheating after cold-beating shown by copper and steels. P. NICOLAU (*Compt. rend.*, 1928, 186, 696–699).—The curves relating the hardness (Brinell) and the temperature of reheating of copper bars (preheated to 700° and cold-beaten to the extent of 5–660%) show systematic anomalies after a preliminary “germination” period, during which the internal equilibrium destroyed by the cold-beating is gradually restored, corresponding with a minimum hardness. Alpha-steels show points of inflexion, and the internal tensions revealed after immersion in a 0.15% aqueous solution of mercuric chloride disappear at reheating temperatures corresponding with these points.

J. GRANT.

Electrical properties of ferronickels containing added chromium. P. CHEVENARD (*Compt. rend.*, 1928, 186, 431–433).—The author summarises the results of his researches. The allotropic transformation of iron-nickel alloys, rich in iron, is accompanied by an increase or decrease in resistivity on heating or cooling, respectively, to an extent increasing with the nickel content up to 30%, and then decreasing till it becomes zero for the compound Fe_3Ni (34.45% Ni). The transformation of alloys rich in chromium and poor in nickel from the α - to the γ -state is marked by a drop in resistivity. The reversible magnetic transformation of austenitic alloys is shown as a break on the resistivity-temperature curve, and decreases in intensity with the nickel and chromium contents. The addition of chromium lowers the temperature of the transformation. Between 500° and 550° paramagnetic chromium ferronickels show a second, imperfectly reversible transformation, the temperature of which is independent of the composition of the alloy. The curves representing the thermo-electric properties of ferronickels as a function of temperature are analogous.

J. GRANT.

Causes of the variation in volume accompanying the hardening of light aluminium-copper alloys. P. CHEVENARD and A. PORTEVIN (*Compt. rend.*, 1928, 186, 144–146).—The effect of the temperature of reheating on the change in length of an aluminium alloy containing 4.30% Cu, 0.80% Mn, and 0.38% Si has been determined, the alloy being previously tempered. The diminution in dilatability is an approximate measure of the Al_2Cu precipitated. Above 170° the curves conform to the authors' theory (B., 1923, 391 A) of the precipitation of Al_2Cu , but below 170° another reaction, accompanied by a diminution in volume, exists with the first. This is attributed to the presence

of impurities rather than to the production of a state analogous to the martensitic structure of steel.

J. GRANT.

Rapid analysis of ordinary brass and red brass. W. KOLLREPP (Chem.-Ztg., 1928, 52, 183).—A technical method for the rapid analysis of brass free from manganese and phosphorus is as follows: 1 g. of turnings is dissolved in 10 c.c. of water and 15 c.c. of nitric acid, diluted with 20 c.c. of hot water, filtered, washed, and the residue of tin oxide ignited and weighed. 10 c.c. of sulphuric acid (d 1.84) are added to the filtrate, which is kept for 15 min. at 60° and the precipitated lead sulphate collected on a filter, ignited, and weighed. The filtrate is neutralised with ammonia, acidified with hydrochloric acid, and the boiling solution treated with hydrogen sulphide. The copper sulphide is removed by filtration and neglected, the filtrate boiled to expel hydrogen sulphide, oxidised with 5 c.c. of nitric acid (d 1.4), excess of ammonia added, and the whole boiled. The precipitated ferric hydroxide is collected on a filter, washed, and weighed as ferric oxide. Nickel is determined in the filtrate by the glyoxime method, and, after removal, zinc is precipitated with hydrogen sulphide and weighed as the oxide. Copper is determined by difference.

C. J. SMITHELLS.

Action of hydrochloric acid on extra-pure aluminium. J. CALVET (Compt. rend., 1928, 186, 369—371).—The resistance to the action of 2.5*N*-hydrochloric acid at 18° previously recorded for extra-pure aluminium (cf. Matignon and Calvet, A., 1928, 251) is only temporary, and after a few days the metal is attacked at a rate which increases with the time and finally remains constant. The period of passivity depends on the origin of the aluminium and on the impurities it contains, and is prolonged if the metal is in a laminated condition. The rate of attack was measured by the volume of gas evolved per sq. cm. of surface.

J. GRANT.

Corrosion of aluminium. W. GUERTLER (Z. Metallk., 1928, 20, 104—112).—A review of the chief factors influencing the rate of corrosion of aluminium and of the mechanism of corrosion phenomena in aluminium. Inclusions of coarse particles of silicon, FeAl_3 , and non-metallic impurities cause the protective layer of oxide to break, thus allowing penetration into the metal of corroding media. During rolling or drawing these inclusions tend to break up and may cause scaling on the surface or minute surface fissures. Trouble due to this cause may be averted by annealing at 400°, followed by quenching, whereby the silicon and iron are retained in solid solution or caused to separate in a finely-divided form. Care should be taken in working and annealing the metal not to develop a coarsely crystalline structure which tends to cause a break-up of the protective skin of oxide.

A. R. POWELL.

Destruction of aluminium by mercury and means for preventing it. H. RÖHRIG (Korrosion u. Metallschutz, 1927, 3, 121—123; Chem. Zentr., 1927, II, 974).—The amalgamation of aluminium vessels by mercury causes rapid destruction of the vessel. Heating the spot removes the mercury effectively, but there is great danger of the vapour condensing on other parts; treatment with concentrated nitric acid, which dissolves

the mercury without attacking the aluminium, is satisfactory for thick-walled vessels, but the safest procedure consists in rubbing the spot with a warm 10% solution of potassium dichromate which, by converting the mercury into a brown oxide or basic chromate, allows it to be readily rubbed off.

A. R. POWELL.

Light metals and alloys. Aluminium. Magnesium (U.S. Bur. Stand., Circ. 346. 403 pp.).—A review of the physical properties of aluminium and magnesium and their alloys.

Determination of fluorine in zinc blende. L. FRESSENIUS, K. SCHRÖDER, and M. FROMMES (Z. anal. Chem., 1928, 73, 65—69).—The finely-powdered ore (2 g.) is fused with 10 g. of a mixture of equal parts of sodium carbonate and peroxide, the product leached with water, and the solution treated with 8 g. of ammonium carbonate, evaporated to 50 c.c., and filtered. The cold filtrate is acidified with 1:1 sulphuric acid and 10 c.c. excess acid are added, followed by 5 c.c. of 3% hydrogen peroxide and 5 c.c. of a standard titanium sulphate solution (1 c.c. = 1 mg. of titania). The mixture is then diluted to 150 c.c. in a white porcelain basin. A similar solution is prepared using the same reagents but without addition of the ore; to this solution is added a solution of sodium fluoride (1 c.c. = 1 mg. of fluorine) until the colour has faded to match that of the assay. The burette reading in c.c. gives directly the number of mg. of fluorine in 2 g. of the blende.

A. R. POWELL.

Composition of old Roman lead. W. A. COWAN (Inst. Metals, March, 1928. Advance copy. 2 pp.).—A description and analysis of an old Roman lead pipe, dating from A.D. 69—79. The lead contained 0.00785% Ag, 0.0328% Bi, 0.0060% Cu, 0.0004% Fe, and a trace of antimony, but no arsenic, tin, or cadmium. The pipe was found in Rome, but is of much the same composition as that previously reported (Friend and Thorneycroft, B., 1927, 281) for a pipe found in England.

W. HUME-ROTHERY.

Historical note on density changes caused by the cold-working of metals. H. O'NEILL (Inst. Metals, March, 1928. Advance copy. 3 pp.).—The decrease in density produced by the cold-working of metals was probably known to Berzelius in 1844, but a detailed paper on the changes in density of copper when reduced by hammering was published by C. O'Neill in 1861 (Mem. Manchester Phil., 1861, 1, [iii], 243).

W. HUME-ROTHERY.

Analytical Commission of the Platinum Institute. I. Reception of platinum ore. II. Rapid analysis of platinum ore. S. F. SHEMTSCHUSHNI, O. E. ZVJAGINSTSEV, B. G. KARPOV, V. V. LEBEDINSKI, and N. I. PODKOPEV. III. Analysis of platinum ores. IV. Determination of copper and iron. V. Complete analysis of platinum ore. A. T. GRIGORIEV, S. F. SHEMTSCHUSHNI, O. E. ZVJAGINSTSEV, B. G. KARPOV, N. S. KURNAKOV, V. V. LEBEDINSKI, and N. I. PODKOPEV. VI. Analysis of the first insoluble residue obtained on dissolving platinum ore with aqua regia. S. F. SHEMTSCHUSHNI, O. E. ZVJAGINSTSEV, B. G. KARPOV, V. V. LEBEDINSKI, and N. I. PODKOPEV (Ann. Inst. Platine, 1926, No. 4, 339, 340—

343, 343—355, 355—359).—I. The ore (2 g.) is melted with silver (7—10 g.) and a flux to remove vanadium, tungsten, iron, etc. Tungsten is extracted from another portion of the ore by digesting it in a mixture of hydrofluoric and nitric acids; the solution is evaporated to recover tungsten trioxide.

V. The mineral is dissolved in aqua regia, the liquid filtered, evaporated with hydrochloric acid, and diluted; chlorine is passed in at 40° to form iridium tetrachloride and prevent reduction of the gold, the mixture evaporated at 38—42°, diluted, and precipitated at 30° with ammonium chloride, the precipitate containing platinum and iridium. Boiling aqueous dimethylglyoxime is added to a portion of the filtrate, gold and palladium being precipitated. The filtrate from this is reduced with zinc and hydrochloric acid, with precipitation of copper, rhodium, and the remaining iridium. Iron is precipitated as hydroxide from the last filtrate after oxidation. The main portion of the filtrate from the precipitation of platinum and iridium is treated with concentrated nitric acid to decompose the ammonium chloride; the residue is dissolved in water and digested at 80—100° with alkaline sodium nitrite. The precipitate consists of gold and base metals. The filtrate, containing rhodium, iridium, ruthenium, platinum, and palladium, is evaporated with hydrochloric acid to remove the nitric acid, the palladium precipitated with mercuric cyanide, the remainder of the platinum and iridium with ammonium chloride, and then all the metals with zinc. The precipitate is redissolved in aqua regia, the iridium precipitated with ammonium chloride, and the filtrate again reduced with zinc to obtain rhodium. All iridium precipitates are reduced to the metal, which is heated in fused sodium carbonate and nitrate and the product is dissolved in hydrochloric acid, which dissolves the ruthenium, leaving a residue of iridium oxide. The ruthenium is recovered by precipitation with zinc.

VI. The precipitate is treated with ammoniacal ammonium acetate solution to remove lead sulphate and silver chloride, and then fused with borax and precipitated silver. The silver button is boiled with sulphuric acid to dissolve the silver, and the residue of osmiridium is fused with zinc in an atmosphere of hydrogen or under a mixture of potassium and sodium chlorides. The mass is dissolved in water, nitric and hydrochloric acids are added, and osmium tetroxide is removed by distillation. Excess of sodium hydroxide is added to the remaining solution, and the ruthenium distilled off in a stream of chlorine, the treatment being repeated after addition of further quantities of sodium hydroxide; the ruthenium is recovered from the distillate by treatment with magnesium. The solution in the distillation flask is acidified and filtered, the filtrate oxidised with chlorine and precipitated with ammonium chloride, and further treated as in the general method for small amounts of iridium, palladium [? platinum], and (in the final filtrate) rhodium; these are precipitated with zinc, ignited, reduced, and cooled in a stream of carbon dioxide.

CHEMICAL ABSTRACTS.

Fatigue produced [in metals] by prolonged application of a constant load. G. WELTER (Z.

Metallk., 1928, 20, 51—57).—The behaviour of bars of metals suspended at one end and loaded at the other with a weight equivalent to 10—20% less than the breaking strength of the metal has been investigated. With a load 10% below the breaking strain, aluminium broke in 3 days, copper in 1 min., brass in 26 hrs., and duralumin in 24 hrs.; the corresponding values for a load 20% below the breaking strain were 8 months, 9 months, 25 days, and infinity. The degree of bending of bars of all the above-named metals, as well as of annealed soft iron, copper, and aluminium, increases rapidly during the first few days under a load equivalent to the limit of proportionality, then proceeds at a small but more or less constant rate over a period of months. With a load equivalent to the elastic limit, no bending takes place with brass, duralumin, and soft iron; the bending-time curve of aluminium is a slightly inclined straight line during the first few days, and that of electron a smooth curve becoming a straight line. With the same load, the bending-time curve taken over a period of months rises steeply concave to the time axis for electron and for annealed copper and aluminium. These results show that iron, brass, and duralumin are safe materials for constructional purposes as long as the load to which they are subjected does not exceed the elastic limit, whereas soft aluminium, copper, and electron undergo progressive fatigue with such a load.

A. R. POWELL.

Changes in the mechanical properties [of metals] caused by fatigue. J. CZOCHRALESKI and E. HENKEL (Z. Metallk., 1928, 20, 58—62).—Test-pieces of steel, aluminium, and copper in the cold-worked and in the annealed state were subjected to fatigue induced by rotating them in a horizontal position in a machine while applying a downward pull to the bearings holding each end of the specimen. The number of reversals required to produce fracture under varying loads was determined as well as the alteration induced in specimens treated in this way with loads equal to and slightly exceeding the elastic limit. In the case of annealed aluminium, the breaking strain is practically unaffected by this treatment, whereas that of copper increases, and that of steel decreases with the load applied; in all cases, however, the elastic limit and limit of proportionality increase with the load applied in the fatigue treatment within the critical limit. With hard-drawn metals the change of mechanical properties is irregular, but not so great as with the annealed metal. With 1 million reversals the critical fatigue load of soft aluminium is 4—5 kg./mm.², of hard-drawn aluminium 6—7 kg./mm.²; the corresponding values for copper are 8—10 and 16—18 kg./mm.², and for steel 20 and 30 kg./mm.². The cylindrical test-pieces on failure in the fatigue test develop cracks on the surface at angles of 45° to the axis in both directions. The cause of these cracks is discussed at some length together with the bearing of the results obtained on the question of the suitability of a metal for constructional purposes.

A. R. POWELL.

Elasticity [of metals]: static and endurance tests. W. KUNTZE, G. SACHS, and H. SIEGLERSCHMIDT (Z. Metallk., 1928, 20, 64—68).—If a metal test-piece is stretched in the testing machine and then subjected to a gradual application and removal of load nearly

up to its new elastic limit the curves showing the elongation with application and removal of load do not coincide, although there is no permanent elongation. The elongation-load curve is a straight line up to $\frac{2}{3}$ — $\frac{3}{4}$ of the elastic limit, then curves away from the load axis; for loads above this point, *i.e.*, on the curved section, the hysteresis during removal of the load is relatively great, and a small deformation remains for some considerable time after complete removal of the load. Endurance tests appear to show that this slight semi-permanent internal stress alone is not sufficient to cause the breakdown of the metal. Alternating bending tests of thick wires on sharp edges give results which seem to bear some relation to the reduction of area obtained in the ordinary tensile tests, but the values obtained for breaking similar wires by alternately bending them round two rolls bear a definite relation to the tensile strength, *i.e.*, to the resistance to deformation. A. R. POWELL.

Fatigue in single crystals [of metals]. E. SCHMID (Z. Metallk., 1928, 20, 69—74).—The tensile strength of a single-crystal wire of zinc is considerably increased by subjecting the wire to a repetition of twists through a small angle, whilst the ductility and bending strength are greatly reduced. As the number of twists is increased the load required to cause the wire to stretch is rapidly increased and the inclination of the load-elongation (within the plastic range) curve becomes smaller. The metal, however, becomes exceedingly brittle, and after relatively few twists breaks along the basal plane when subjected to even a small bending stress without undergoing any plastic deformation. These results confirm the work of Gough, Hanson, and Wright on aluminium single-crystal wire (A., 1926, 666, 997).

A. R. POWELL.

Quantitative measurement of the cutting power of cutlery. K. HONDA and K. TAKAHASHI (Sci. Rep. Tôhoku Imp. Univ., 1927, 16, 755—773).—See B., 1927, 782.

Blasting in metal-mine drifts. GARDNER and others.—See XXII.

PATENTS.

Production of cast-iron castings by a centrifugal process. NEWTON CHAMBERS & Co., LTD., and J. E. HURST (B.P. 285,721, 15.7.27).—The structure of centrifugal castings of cast iron is rendered sorbitic by keeping the silicon content to 1.25—2.5% when metal moulds are used and to 0.5—1.5% when sand moulds are used. In either case the castings are caused to cool rapidly by directing a blast of air, preferably moist, on their inner surface. A. R. POWELL.

Hardened cast iron. K. SIPP, Assr. to H. LANZ A.-G. (U.S.P. 1,658,467, 7.2.28. Appl., 3.11.25. Ger., 10.11.24).—Cast iron which has been subjected to the pearlite process (cf. B.P. 147,933 and 210,091; B., 1921, 853 A; 1925, 176) is heated and quenched in the same manner as steel. T. S. WHEELER.

Manufacture of a metal-treating compound. M. L. MOYER, Assr. to M. L. MOYER, E. J. PRINDLE, and B. STROUGHTON (U.S.P. 1,659,214, 14.2.28. Appl., 9.10.25).—Iron is toughened by being cooled in a

mixture of soap, sperm oil, alum, mercuric chloride, zinc sulphate, lead monoxide, and carbon.

T. S. WHEELER.

Iron mould. S. M. UDALE, Assr. to E. HOLLEY (U.S.P. 1,658,144, 7.2.28. Appl., 23.11.25).—A cast, permanent mould for iron is formed of an alloy consisting of 34.5% Ni, 2.0% C (mostly free), 1.3% Si, 0.8% (Mn, S, and P), and the balance iron.

T. S. WHEELER.

Manufacture of [ferrous] alloys. V. B. BROWNE (U.S.P. 1,658,879, 14.2.28. Appl., 21.10.25).—In the manufacture of alloy steel in an arc furnace, the metal bath is subjected until free from silicon to the action of any usual oxidising slag, the slag is replaced by a protective layer of calcium oxide, and the metal freed from oxides by addition of aluminium.

T. S. WHEELER.

Recovery of metals [copper, zinc, etc.] from iron pyrites and similar ores and from liquors containing them. C. F. SCHANTZ (B.P. 285,662, 12.3.27).—Leach liquors containing copper and zinc derived from sulphide ores which have been subjected to a chloridising roast are treated with sulphuric acid equivalent to their chlorine content, which is then precipitated as cuprous chloride by the addition of the necessary quantity of sponge copper and cupric oxide; at the same time the whole of the lead is deposited as sulphate and the silver and gold in the metallic form, leaving a solution containing sodium and zinc sulphates. After further purification the clear solution may be used for the manufacture of lithopone, zinc white, or electrolytic zinc; in the first two cases sodium sulphate may be recovered from the filtrates, and in the third case the acid liquor obtained is used in the first stage of the process. The cuprous chloride precipitate is extracted with a concentrated alkali chloride solution and the copper recovered from the resulting solution by cementation on iron or by electrolysis; the insoluble residue is worked up for its content of lead and precious metals by known methods. The spent electrolyte from the copper cells is used for extracting further quantities of cuprous chloride sludge. Instead of using an outside source of current for the deposition of copper, this may be effected by placing a sheet of copper in the solution and joining it outside the liquor to a sheet of iron immersed in dilute sulphuric acid in a porous cell which is placed in the cuprous chloride solution; ferric sulphate is obtained by evaporating the liquor in the cell after use.

A. R. POWELL.

Zinc-base alloy. W. MCG. PEIRCE and E. A. ANDERSON, Assrs. to NEW JERSEY ZINC Co. (U.S.P. 1,663,215, 20.3.28. Appl., 5.1.27).—Zinc is alloyed with 1—15% Al, 0.5—4% Cu, 0.05—5% Mg, and 0.01—0.5% Ni.

F. G. CROSSE.

Refining metals [removal of zinc from lead]. E. C. R. MARKS. From AMER. SMELTING & REFINING Co. (B.P. 285,630, 21.1.27).—Molten lead from the Parkes process is dezincified by pumping it continuously in a fine stream through a closed cylinder filled with chlorine, in such a way that a thin layer of lead is maintained on the floor of the cylinder while the treated lead passes continuously back into the bottom of the kettle containing

the bulk of the metal. The zinc chloride formed in the early stages of the process is free of lead and may be rejected, but that from the later stages contains much lead and is therefore used in the preliminary treatment of a fresh kettle of metal. The zinc content of the lead may be rapidly reduced to less than 0.003% by this process.

A. R. POWELL.

Aluminium-base alloy. R. S. ARCHER, Assr. to ALUMINUM Co. OF AMERICA (U.S.P. 1,663,150, 20.3.28. Appl., 30.6.25).—Aluminium is alloyed with 3–15% Si, more than 0.8% Fe, and more than 0.05% of a metal of the sixth group of the periodic system having an at. wt. less than 190.

F. G. CROSSE.

Bending of section rods of magnesium alloys. I. G. FARBERIND. A.-G. (B.P. 284,313, 5.1.28. Ger., 28.1.27).—The rods are heated at 100–400° before being passed through bending rollers.

H. ROYAL-DAWSON.

Colouring of [tin] foil. W. G. GOLDEN, Assr. to U.S. FOIL Co. (U.S.P. 1,662,574, 13.3.28. Appl., 26.10.25).—A gold colour is imparted to tin foil by the direct application of heat.

H. ROYAL-DAWSON.

Soldering material. S. RUBEN (U.S.P. 1,663,141, 20.3.28. Appl., 1.10.26).—An alloy of 34% Pb, 34% Sn, 31% Zn, and 1% Mg is claimed.

F. G. CROSSE.

Cyaniding of ores containing precious metals. S. B. McCLUSKEY (U.S.P. 1,658,249, 7.2.28. Appl., 8.10.26).—The pulp mixed with cyanide solution is treated with sulphur dioxide, and the hydrogen cyanide liberated is volatilised. Air is blown through the mixture to oxidise ferrous and manganous salts, which are then precipitated by addition of calcium hydroxide and carbonate. The pulp is next treated with the hydrogen cyanide evolved in the first stage, cyaniding now proceeding smoothly in the absence of base-metal salts.

T. S. WHEELER.

Concentration of ores and minerals by flotation. W. A. DOUGLASS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,659,396, 14.2.28. Appl., 25.10.26).—An organic derivative of trithiocarbonic acid is claimed as a flotation reagent.

T. S. WHEELER.

Production of rare-metal powders. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,659,209, 14.2.28. Appl., 3.1.23).—In the process of U.S.P. 1,602,542 (B., 1927, 46) calcium chloride is added as a flux to the mixture of rare metal oxide and magnesium.

T. S. WHEELER.

Cleaning and coating of metal articles. "METALLOGEN" G.M.B.H. (B.P. 285,556, 16.11.26. Addn. to B.P. 154,235; B., 1921, 87A).—An apparatus for carrying out the process of the chief patent is described.

A. R. POWELL.

Manufacture of steel and alloys from iron sand. D. CROESE (U.S.P. 1,662,302, 13.3.28. Appl., 19.2.26. U.K., 26.2.25).—See B.P. 252,455; B., 1926, 674.

Production and treatment of a copper alloy. M. G. CORSON, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,658,186, 7.2.28. Appl., 25.9.25. Can., 21.2.25).—See B.P. 256,457; B., 1926, 885.

Bearing composition. H. M. WILLIAMS, Assr. to GEN. MOTORS RES. CORP. (U.S.P. 1,661,245, 6.3.28. Appl., 22.5.23).—See B.P. 216,484; B., 1924, 1017.

XI.—ELECTROTECHNICS.

Theory of magnetic separation. B. W. HOLMAN (Trans. Inst. Chem. Eng., Advance Proofs, March, 1928; 3–11).—Theories of magnetism are briefly discussed in relation to the operation and design of magnetic separators, particular consideration being given to the influence of particle size, the shape of the pole pieces, the design of electro-magnets, the use of flux-meters, and the determination of permeability.

A. B. MANNING.

Measurement of the electrical resistance of porous materials. A. E. KNOWLER (Proc. Physical Soc., 1928, 40, 37–40).—The resistance of a specimen of refractory material is determined by passing a known current through and measuring the potential between two wires inserted in it a known distance apart. The method avoids errors due to contact resistances.

C. J. SMITHELLS.

Electrical insulating materials. O. KRAUSE (Z. Elektrochem., 1928, 34, 49; cf. Günther-Schulze, B., 1927, 881).—Polemical.

H. J. T. ELLINGHAM.

Electro-osmosis of water. PATIN. Removal of oil from condenser water. RÂSCANU.—See XXIII.

PATENTS.

Electrolytic rectifier. A. B. MUNTAN, Assr. to G. E. FISCHER (U.S.P. 1,662,075, 13.3.28. Appl., 15.10.24. Holl., 19.10.23).—Aluminium anode and cathode members, previously coated with different thicknesses of metallic oxide, are immersed in an electrolyte, and, when so immersed, have a constant difference of electrical capacity per unit area from one another, possess the correct capacities in reference to the current to be rectified, and remain unaltered by operation of the rectifier.

J. S. G. THOMAS.

Electrolytic rectifier. W. H. GRINDITCH and J. N. HUNSBARGER, JUN., Assrs. to PHILADELPHIA STORAGE BATTERY Co. (U.S.P. 1,662,383, 13.3.28. Appl., 18.2.27).—A rectifying electrode for asymmetric cells consists of aluminium containing 0.04–0.4% Cu.

J. S. G. THOMAS.

Salt for electrolytic rectifiers. F. W. BARHOFF and W. C. BROOKS, Assrs. to HARTFORD BATTERY MANUF. Co. (U.S.P. 1,658,914, 14.2.28. Appl., 26.5.27).—A mixture of ammonium sulphate, sulphuric acid, and ferrous sulphate forms a solid product which dissolves in water to yield an electrolyte suitable for use with filming electrodes of tantalum.

T. S. WHEELER.

Electrolyser resembling a filter press [for the electrolysis of water]. A. E. ZDANSKI (Swiss P. 120,249, 4.1.26. Ger., 25.3.25).—An apparatus for the decomposition of water comprises a deep cell with a series of compound electrodes arranged one above the other and resembling in structure a filter press. The potential of the electrodes increases with their height above ground.

A. R. POWELL.

Electrolysis of salt solutions. DEUTS. GAS-GLÜHLICHT-AUER-GES.M.B.H. (G.P. 446,009, 27.8.24).—

An apparatus for the purpose comprises a flat cell with an internal inclined, water-cooled false bottom acting as one electrode, and a superimposed chamber with diaphragms inside which is a second flat electrode perforated with inclined slits. A. R. POWELL.

Art of incrusting galvanoplasty. F. MAAS, Assr. to NATURAL FLOWER METALIZING CO., INC. (U.S.P. 1,662,430, 13.3.28. Appl., 26.8.25).—An object is coated, in succession, with stiffening material, with a base containing phosphorus, rubber, asphaltum, carbon disulphide, and binding material, with silver nitrate, and is then electroplated. J. S. G. THOMAS.

Metallic cores for electromagnets etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 286,167, 5.9.27. Addn. to B.P. 269,770; B., 1927, 493).—The metallic powder produced from the carbonyl is heated in an atmosphere of reducing gas (e.g., 18 hrs. at 500° in a current of hydrogen) prior to being made into cores. J. S. G. THOMAS.

Cleaning-up of residual gases [in thermionic valves]. A. E. LYLE, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,659,207, 14.2.28. Appl., 7.11.24).—Small quantities of lime and magnesium are introduced into a thermionic valve containing a filament coated with an alkaline-earth carbonate. The tube is evacuated and the filament heated to decompose the carbonate to the oxide, the carbon dioxide evolved being absorbed by the lime. The magnesium metal is then heated by induction to complete gettering. T. S. WHEELER.

Electric resistance furnace adapted for heating by nitrogenation. A. FRY, Assr. to F. KRUPP A.-G. (U.S.P. 1,661,694, 6.3.28. Appl., 2.8.26. Ger., 4.9.25).—See B.P. 257,896; B., 1927, 786.

Manufacture of a tungsten product [filament]. A. DE GRAAFF, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,662,027, 6.3.28. Appl., 24.4.24. Holl., 17.5.23).—See B.P. 216,124; B., 1925, 91.

Arrangements for feeding [electric] furnaces working with open arcs. A.-G. BROWN, BOVERI, & Cie. (B.P. 261,785 and 263,118, [A] 20.11.26, [B] 29.11.26. Switz., [A] 21.11.25, [B] 15.12.25).

XII.—FATS; OILS; WAXES.

Action of solutions of potassium iodate and hydrogen iodide on fats. B. M. MARGOSCHES, K. FUCHS, and B. KRAKOWETZ (J. pr. Chem., 1928, [ii], 118, 225—237).—The fat is treated with 0.2*N*-aqueous hydriodic acid (which may be preserved unchanged for some 3 weeks) and excess of potassium iodate in aqueous alcoholic solution (cf. the "rapid iodine-number method," B., 1924, 341, 639). When the mixture is immediately titrated with thiosulphate, iodine numbers are obtained which, after doubling (owing to reoxidation by the iodate of the hydriodic acid formed in the reaction, $I_2 + H_2O = HI + HIO$), agree closely with the Hübl or Hanus values. After 5 min. the mixture gives a lower value. This decrease, which continues for some 24 hrs., is probably due to reactions similar to those connected with the iodine super-number (A., 1925, i, 629, 833). When equivalent quantities of hydriodic acid and

potassium iodate are used the iodine absorption after 5 min. corresponds with that obtained by the rapid method. Castor oil and resins which liberate more than 50% of free acid in the rapid method (cf. A., 1924, i, 828) naturally give low iodine numbers in presence of excess of iodate. H. E. F. NOTTON.

Comparison of the methods for separating solid and liquid fatty acids. K. AMBERGER and E. WHEELER-HILL (Z. Unters. Lebensm., 1927, 54, 431—434).—The methods compared were (1) the insolubility of the lead salts of the solid fatty acids in ether, (2) Twitchell's process (B., 1921, 817 A), and (3) the thallium process as modified by Holde (B., 1924, 755). The first process was found unsuitable for quantitative work as the solid acids recovered still contained appreciable proportions of liquid acids. The other two processes gave quantitative separations of palmitic acid from oleic and linoleic acids. Neither process was satisfactory with erucic acid. J. R. NICHOLLS.

Fatty acids of seed oils of *Brassica* species. Composition of rape, ravison, and mustard seed oils. T. P. HILDITCH, T. RILEY, and N. L. VIDYARTHI (J.S.C.I., 1927, 46, 457—462 r).—The composition of the fatty acids present as glycerides in four representative fats of the *Brassica* family, viz., English rape, Danubian ravison, and English black and white mustard seed oils, has been investigated. The procedure consisted in effecting a partial separation of the fatty acids into three groups, according to the solubility of their lead salts in alcohol and ether, followed by systematic fractional distillation of the methyl esters of each group of acids under reduced pressure. It was found that each of the fats was composed, qualitatively, of the same acids. The main components were erucic acid (about 50% of the whole) and unsaturated acids containing 18 carbon atoms (about 45—47%); the latter consisted for the most part of ordinary $\Delta^9:10$ -oleic acid and linoleic acid (the relative proportions of these varying considerably), together with small quantities of linolenic acid. There was also present in each case a small amount of isomeric oleic acid (or acids), which separated with the least soluble portion of the lead salts, and which yielded on oxidation a dihydroxy-compound (m.p. 117—118°), which was differentiated from the other known dihydroxystearic acids by its relatively great solubility in ether, alcohol, and ethyl acetate. The remaining fatty acids present belonged to the saturated series, and included 1—2% each of palmitic and lignoceric acids, with traces of stearic and arachidic acids. The quantitative results obtained by the systematic fractionation of the esters are given in the following table:—

Oil.	English rape seed.	Danubian ravison.	English black mustard (charlock).	English white mustard.
Source:	<i>Brassica campestris</i> .	<i>B. campestris</i> var.	<i>B. (Sinapis) nigra</i> .	<i>B. (S.) alba</i> .
Combined fatty acids.	%	%	%	%
Palmitic	1	2	2	2
Stearic	—	—	Trace	Trace
Arachidic	—	—	Trace	1
Behenic	—	Trace	—	—
Lignoceric	1	2	2	1
Oleic	32	20.5	24.5	28
Linoleic	15	25.5	19.5	14.5
Linolenic	1	2	2	1
Erucic	50	47	50	52.5

III-defined acids of the oleic series. III. "Rapic acid" and other acids of rape and mustard seed oils. T. P. HILDITCH, T. RILEY, and N. L. VIDYARTHI (J.S.C.I., 1927, 46, 462—467 r). The fatty acids present in the four *Brassica* fats dealt with in the preceding paper (see above) have been submitted to a detailed examination, mainly in order finally to settle whether any isomerides of oleic acid are present (e.g., the "rapic acid" of Reimer and Will and of Zellner, the existence of which was denied by Grabner, Raymond, and Toyama). Quantitative oxidation of the methyl esters of the unsaturated C_{18} acids (the lead salts of which are soluble in alcohol or ether) has established that they are derived practically exclusively from acids containing an ethylenic linkage in the $\Delta^{9:10}$ position (ordinary oleic and linoleic acids). Unsaturated C_{18} acids separated in the form of insoluble lead salts constitute a relatively small proportion of the whole, and of these, probably about half is also the ordinary $\Delta^{9:10}$ -oleic acid. Consequently isomeric forms are only present to the extent of 1—2% at most of the total combined fatty acids of these oils. On the other hand, the presence of the 1—2% of isomeric oleic acid (or acids) is readily distinguished in the *Brassica* fats by the formation, from the unsaturated acids of the C_{18} series which form insoluble lead salts, of the unusually soluble dihydroxy-compound which melts at 117—118° (cf. preceding abstract). By systematic lead salt separation and fractionation of a large quantity of rapeseed oil fatty acids sufficient of the isomeric acid (still admixed with some ordinary oleic acid) was finally obtained for oxidation of the corresponding methyl ester in acetone solution with potassium permanganate. The resulting acidic products (in addition to azelaic acid and nonoic acid from ordinary oleic acid) included a solid dibasic acid (which melted at 137—138°, and corresponded with the formula $C_{10}H_{18}O_4$, but was not identical with sebacic acid, $CO_2H \cdot [CH_2]_8 \cdot CO_2H$) and a mixture of monobasic acids in which octoic and decaic acids were apparently both present. It was therefore concluded that, although Reimer and Will's "rapic acid" is certainly non-existent in the sense indicated by these workers, there is present in the fats examined a small amount of acid $C_{18}H_{34}O_2$; most of this is probably a branched-chain unsaturated acid of the general structure $C_7H_{15} \cdot CH : CH \cdot (C_8H_{16}) \cdot CO_2H$, but more than one individual acid is probably present. The compounds in question are referred to as "isoleic acids of the *Cruciferae*." The erucic acid, $C_{22}H_{42}O_2$, present in these fats was shown to be exclusively $\Delta^{13:14}$ -dodecenoic acid, and no indication was obtained of the presence of isomeric forms similar to those of the oleic acids of this group. The lower saturated acid present in the fats examined was almost entirely palmitic acid; myristic acid cannot have been present in amounts exceeding about 0.2%.

III-defined acids of the oleic series. IV. "Cheiranthic acid" of wallflower seed oil. T. P. HILDITCH and (Miss) E. E. JONES (J.S.C.I., 1927, 46, 467—469 r).—The combined fatty acids of wallflower seed oil, stated by Matthes and Boltze to consist of 65% of cheiranthic acid, $C_{18}H_{34}O_2$, 30% of linoleic acid, and 5% of linolenic acid, have been shown (by separation into three groups

of fatty acids by lead salt separation, followed by systematic fractionation of the methyl esters of each group) to consist of: palmitic (3%), lignoceric (0.5%), ordinary oleic (12.5%), linoleic (41%), linolenic (4%), and erucic (39%) acids. Wallflower seed oil is, therefore, of the same general type as rape, mustard, and other seed fats of the same botanical order, *Cruciferae*. The only marked differences are a lower content than the average of combined erucic acid, and the much less saturated nature of the acids containing 18 carbon atoms compared with those in the other *Cruciferae* fats so far examined. "Cheiranthic acid" as an individual isomeric form of ordinary oleic acid is a case of mistaken identity; it should be removed from the list of naturally-occurring unsaturated fatty acids.

Head and blubber oils of the sperm whale. I. Determinations of the mixed fatty acids present. T. P. HILDITCH and J. A. LOVERN (J.S.C.I., 1928, 47, 105—111 r).—The head and blubber oils of the Antarctic sperm whale examined consisted of a mixture of higher fatty alcohol esters and glycerides of fatty acids, the wax or ester components largely predominating (head oil, about 74% of wax; blubber oil, about 66% of wax). The head oil is less unsaturated than the blubber oil, and contains larger amounts of compounds of relatively low molecular weight, both as regards the acids and alcohols. The latter appear to consist, roughly, of (i) about 60% of C_{16} alcohols (partly unsaturated), 35% of C_{15} alcohols (mainly unsaturated), and 5% of unsaturated C_{20} alcohols (head oil), and (ii) about 20% of C_{16} alcohols (partly unsaturated), 70% of C_{18} alcohols (largely unsaturated), and 10% of unsaturated C_{20} alcohols (blubber oil); the composition of the alcohols is receiving further study. The mixed acids present have been determined to consist of the following: (i) Head oil: Capric (3.5%), lauric (16%), myristic (14%), palmitic (8%), stearic (2%); dodecenoic (4%), tetradecenoic (14%), hexadecenoic (15%), oleic (17%), and eicosenoic (6.5%); all of the unsaturated acids appear to be monoethylenic. (ii) Blubber oil: Lauric (1%), myristic (5%), palmitic (6.5%); tetradecenoic (4%), hexadecenoic (26.5%), C_{18} acids (average unsaturation — 2.1H) (37%), C_{20} acids (— 2.6H) (19%), and C_{22} acids (?) (— 3.6H) (1%). The wide differences between the fatty acids of the head and blubber oil, and between both types of sperm fatty acids and those of other whale oils and of fish oils, are emphasised.

Composition of oat oil. K. AMBERGER and E. WHEELER-HILL (Z. Unters. Lebensm., 1927, 54, 417—431).—The oil was extracted by keeping the oats for 7 days in light petroleum, and was purified by dissolution in acetone and light petroleum and by washing with water. The fatty acids were separated into palmitic acid (10.4%), α -linoleic acid (17.2%), β -linoleic acid (13.9%), and oleic acid (58.5%). Erucic and linolenic acids were not detected. Individual glycerides were sought from the hardened fat, through the elaidin reaction, and from the brominated oil, and triolein and α -palmito- α -distearin were isolated (cf. Amberger and Bromig, B., 1922, 675 a). J. R. NICHOLLS.

Composition of ivy seed oil. A. STEGER and J. VAN LOON (Rec. trav. chim., 1928, 47, 471—476).—Ivy seed

oil, d_{25}^{25} 0.9151, acid value 11.0, saponif. value 181.1, iodine value 102.2, setting point 13.8°, n_D^{20} 1.4670, obtained in 20% yield from the dried seeds has the following percentage composition; glycerol as C_3H_2 (4.0), unsaponifiable matter (6.6), higher saturated fatty acids (4.5), linoleic acid (11.7), 9:10-oleic acid (17), and petroselinic acid (55). H. BURTON.

PATENTS.

Washing, wetting, and cleaning agents. P. FRIESENHALM (B.P. 269,134, 26.11.26. Ger., 6.4.26).—Hydrocarbons and other solvents are rendered water-soluble by treatment with the soaps and soap-like compounds described in G.P. 365,160 (B., 1923, 318 A), the cyclohexanols in which may be replaced by alcohols or phenols of high mol. wt., alkyl naphthols, etc. On treatment of their aqueous solutions with inorganic salts (e.g., sodium carbonate or sulphate) a solid product separates which contains the salt and the organic solvent.

B. FULLMAN.

Edible fat. H. YUDOWITCH (H. A. NEWTON). (Re-issue 16,915, 20.3.28, of U.S.P. 1,601,229, 28.9.26).—See B.P. 284,368; B., 1928, 284.

Production of emulsions. W. A. WHATMOUGH (U.S.P. 1,663,323, 20.3.28. Appl., 14.5.27. U.K., 13.5.26).—See B.P. 280,096; B., 1928, 99.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

"Accelerated testing" of paints. K. WÜRTH (Farben-Ztg., 1928, 33, 1470).—A brief account of the historical development of accelerated paint tests.

S. S. WOOLF.

Metallic lead in red lead. K. WÜRTH (Farben-Ztg., 1928, 33, 1473—1474).—Heinrich's work on the presence of metallic lead in red lead used in glass manufacture is discussed from the point of view of paint technology.

S. S. WOOLF.

PATENTS.

Production of lithopone. J. E. BOOGE, M. L. HANAHAN, and J. P. KOLLER, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,658,628, 7.2.28. Appl., 23.4.24).—The charge before calcination is screened so as to lie between specified size limits. T. S. WHEELER.

Manufacture of pigments. G. C. LEWIS, Assr. to COLUMBIAN CARBON Co. (U.S.P. 1,659,403, 14.2.28. Appl., 9.9.26).—Humus, e.g., peat, is extracted with sodium hydroxide solution, and the solution treated with acid to precipitate a pigment of value in the manufacture of varnishes.

T. S. WHEELER.

Composition of matter [white pigment]. L. WILLIAMS (U.S.P. 1,662,625, 13.3.28. Appl., 7.5.27).—A mixture of zinc oxide (60 g.), sulphur (2 g.), sodium carbonate (1.5 g.), sodium chloride (1 g.), and ultramarine-blue (0.5.), after being ground, is heated in a suitable closed retort until all the free sulphur has disappeared, and the product is cooled and again pulverised.

H. ROYAL-DAWSON.

Preparation of resin. R. BEUTNER, Assr. to A. NOWACK A.-G. (U.S.P. 1,658,281, 7.2.28. Appl., 15.11.24).—A coal-tar oil is heated with formaldehyde, aqueous sodium hydroxide solution, and kerosene to yield a

product which separates into three layers, comprising, respectively, an oil, an aqueous solution, and a bakelite resin formed from the phenols present in the tar oil.

T. S. WHEELER.

Manufacture of derivatives of the reaction product of amines and aldehydes. C. O. NORTH, Assr. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,659,151, 14.2.28. Appl., 24.3.23).—The products obtained as described in U.S.P. 1,659,152 (see p. 309) are heated with an aldehyde, e.g., formaldehyde, in aqueous solution at about 100° for 6 hrs., and water is removed at 105° to yield resins.

T. S. WHEELER.

Paints, enamels, japons, etc. D. GARDNER (U.S.P. 1,662,741, 13.3.28. Appl., 6.8.22. U.K., 21.8.22).—See B.P. 211,516; B., 1924, 391.

Treatment of dispersed colloids (B.P. 283,686).—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Chemical unsaturation of rubbers vulcanised with polynitro-compounds and benzoyl peroxide, and its possible bearing on vulcanisation. H. L. FISHER and A. E. GRAY (Ind. Eng. Chem., 1928, 20, 294—295).—Samples of rubber vulcanised with dinitrobenzene, trinitrotoluene, and benzoyl peroxide, respectively, were extracted with acetone, dried, and then examined in solution, e.g., in carbon disulphide, as to the degree of chemical unsaturation by means of iodine chloride. The alteration in the degree of unsaturation did not exceed 1%. The results suggest that vulcanisation involves an undetermined type of change in the caoutchouc hydrocarbon without change in its unsaturation.

D. F. TWISS.

Activity of certain aryl-substituted diguanides as accelerators of vulcanisation [of rubber]. G. B. L. SMITH and A. J. WEISS (Ind. Eng. Chem., 1928, 20, 298—300).—Various α -aryldiguanides are compared with several better known accelerators as to their influence on the rate of vulcanisation and the tensile properties in a mixture of rubber 100 pts., zinc oxide 100 pts., sulphur 10 pts., and accelerator 1 pt. The effect of phenyl-, *o*- and *p*-tolyl-, and 3:4-xylyldiguanides compares favourably with that of hexamethylenetetramine, and is therefore of moderate character; $\alpha\alpha$ -diphenyldiguanide is somewhat more active than the monoaryl compounds, but α -naphthyldiguanide is less effective than its monoaryl analogues of lower mol. wt.; with this exception the activity of the monoaryldiguanides increases with the mol. wt. Of the α -tolyldiguanides, the *o*-compound (which was also tested in certain technical rubber mixtures), was more powerful than its *p*-isomeride.

D. F. TWISS.

Comparison of zinc oxide, lithopone, zinc sulphide, and titanium dioxide in [rubber] films vulcanised with sulphur chloride. R. DITMAR and G. BALLOG (Gummi-Ztg., 1928, 42, 1303—1304).—Solutions of masticated pale crêpe rubber (15 pts.) in benzene (100 pts.) were mixed with zinc oxide, lithopone, zinc sulphide, and titanium oxide, respectively, each powder being of colloidal fineness. Dried articles formed by a single immersion of a mould were submitted to

vulcanisation by sulphur chloride in solution or in the vapour state, and were then examined as to physical condition, colour, and ageing in sunlight. With the presence of 8% of each powder (to 100 of rubber), which is the maximum proportion for satisfactory results with such fine ingredients, the zinc oxide was generally most advantageous. D. F. TWISS.

Electrolytic precipitation of fresh and preserved latices and the rôle of the albumin in coagulation. P. SCHOLZ (Kautschuk, 1928, 4, 5—8).—The minimum concentration of an electrolyte, in millimols. per litre, necessary to effect coagulation of latex under standardised conditions is termed the coagulation value of the electrolyte. The coagulation value of chloride of potassium, lithium, barium, or magnesium, or of sulphate of magnesium or nickel, is little influenced by dilution of the latex, but with sulphate of copper, aluminium, or lanthanum, or with acetic acid (which are coagulants for albumin), dilution of the latex causes a marked decrease in the coagulation value; with latex which has been deprived of its natural protein the coagulation value of all these substances is practically unaffected by dilution. The relative behaviour of fresh and preserved and neutralised preserved latex is also investigated. D. F. TWISS.

Stretching of rubber. E. A. HAUSER and P. ROSBAUD (Kautschuk, 1928, 4, 12—14).—With decrease in temperature the stress-strain curve of smooth, unvulcanised, smoked sheet rubber moves nearer to the load axis, and the final extension diminishes; at -185° the rubber is inextensible and the breaking load amounts to 5.45 kg./mm.², which is exceeded only by that of rubber stretched at the ordinary temperature and then cooled in liquid air. Above 40° stretching is accompanied by local constriction with great local extension. The course and length of the stress-strain curve are greatly influenced by the speed of stretching. The proportionality indicated earlier between the intensity of the discontinuous interference in the X-ray spectrum and the heat of extension with the degree of extension holds only for experiments with constant velocity of extension. D. F. TWISS.

Ageing of stretched rubber. A. KELLY, B. S. TAYLOR, and W. N. JONES (Ind. Eng. Chem., 1928, 20, 296—298).—Ageing experiments have been made with various commercial grades of vulcanised rubber, in sunlight, in the Geer oven at 70° , in the Bierer bomb, and in ultra-violet light. Stretching of the test pieces accelerates deterioration, but there is no simple proportionality between the degree of extension and the initial rate of depreciation. In sunlight there is a critical elongation for each sample at which a maximum initial rate of deterioration is obtained. No direct relation is evident between the results of ageing by sunlight and by the other methods. Contrary to the effect of sunlight, ultra-violet light causes a softening of stretched samples. D. F. TWISS.

Effects of ozone on stretched rubber. F. H. HAUSHALTER, W. N. JONES, and J. W. SCHADE (Ind. Eng. Chem., 1928, 20, 300—302; cf. preceding abstract).—Rubber strips stretched even to a small extent in an

atmosphere containing as little as 0.1% of ozone develop cracking in seconds or minutes as compared with weeks in sunlight. In the early stages of exposure, the degree of cracking in ozone, like that in sunlight, increases as the tension is increased up to a certain limit, and beyond that decreases again; in unstretched samples cracking does not occur. D. F. TWISS.

Typical-angle abrasion machine [for rubber]. W. W. VOGT (Ind. Eng. Chem., 1928, 20, 302—306).—A machine is described in which the resistance to abrasion is measured by pressing a ring of the rubber against the flat surface of a driven abrasive wheel in such a way that the plane of the ring makes an angle with the tangent of the abrasive wheel at the point of contact; means are provided for measurement of the power consumed in abrasion. The influence of various factors such as time, speed, load, and angle of contact on the extent of abrasion is discussed, and the mechanical forces involved are analysed. D. F. TWISS.

Theory of needle-shaped caoutchouc molecule in science and practice. E. LINDMAYER (Gummi-Ztg., 1928, 42, 1025—1028).—Various recent investigations and problems are cited, and the possible application of the theory of the needle-shape of the caoutchouc molecule is indicated. D. F. TWISS.

Jelutong. C. D. V. GEORGI (Malayan Agric. J., 1927, 15, 400—407).—Coagulation experiments with sodium silicofluoride, acetic acid, formic acid, sulphuric acid, and potash alum showed the first-named to be superior to the remainder in yielding a product resistant to deterioration by mould development; unfortunately, the toxic properties of sodium silicofluoride prevent its adoption. No tendency to deterioration by "drying out" to a brittle mass was observed even with alcohol as coagulant. Jelutong is preferably coagulated by boiling the acidified latex; thus prepared, it appears to be less porous and to retain its moisture better. D. F. TWISS.

PATENTS.

Treatment of aqueously dispersed colloidal substances. A. BIDDLE (B.P. 283,686, 15.11.26).—Natural or aqueous dispersions, *e.g.*, of rubber, cellulose products, or resins, with or without the addition of oils, waxes, tars, and common fillers, are treated with lime and a reagent, *e.g.*, sodium fluoride, which, by chemical action with the lime, yields an alkali; the nascent condition of the alkali results in a superior product. The lime may be introduced in the form of a compound with a hydrophilic colloid, the latter, if desired, being subsequently rendered insoluble. D. F. TWISS.

Manufacture of the aliphatic aldehyde derivative of the condensation product of an aliphatic aldehyde and an aromatic primary amine. C. O. NORTH, Assr. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,659,152, 14.2.28. Appl., 24.3.23).—A primary arylamine, *e.g.*, aniline, is treated with an aliphatic aldehyde at above 30° to give a compound containing 2 mols. of base to 3 of aldehyde, which is dehydrated at 100° to yield a product of value as an accelerator of vulcanisation. T. S. WHEELER.

XV.—LEATHER; GLUE.

Determination of sugars in tanning materials.

I. D. CLARKE and R. W. FREY (J. Amer. Leather Chem. Assoc., 1928, **23**, 91—108).—Experiments with infusions of different tanning materials and extracts show that, in the determination of sugars, no one period of hydrolysis can be selected for all materials. Infusions of divi-divi pods, wattle bark, and algarobilla pods, respectively, showed the greatest loss of sugary matter with increasing time of hydrolysis. Each tanning material showed a maximum content of sugar after a certain period of hydrolysis. Hydrolysis for only 6 min. gave results 82—100% of the maximum. The operations of clarifying and de-leading introduced salts into the solutions which were thereby buffered and brought to a constant p_H value on the addition of acid, irrespective of the nature of the tanning material. The neutralisation of the hydrolysed solution can be conveniently omitted. Determinations were made of the sugars fermentable and non-fermentable by bakers' yeast in hydrolysed and unhydrolysed solutions. The content (%) of fermentable reducing sugars in chestnut wood, chestnut extract, divi-divi pods, myrobalans nuts, sumac leaves, valonia cups, and wattle bark, respectively, was 40, 37, 88, 85, 83, 85, and 92%; corresponding values for the fermentable, non-reducing sugars are 0, 5, 63, 0, 35, 0, and 36%. Decided differences are shown in the nature of the sugars in the materials. Hydrolysis materially increased the quantity of sugars capable of reducing Fehling's solution, but did not always result in an increase in the amount of fermentable sugars.

D. WOODROFFE.

Molybdenum tannage. J. G. NIEDERCORN (Ind. Eng. Chem., 1928, **20**, 257—258).—A solution of the purple-red sulphate of tervalent molybdenum, prepared by the electrolytic reduction of a solution of molybdic anhydride in sulphuric acid, was used for tanning pelt. It tanned very rapidly at p_H 1—1.5, but no longer penetrated at p_H 2.0. The corresponding green sulphate tanned calfskin at p_H 2—2.5. Both salts yielded a dark brown leather unaltered by boiling. On exposure to air, the leather gradually lost this property, but no hydroxide could be extracted with the boiling water.

D. WOODROFFE.

Progress report on effects of acids on leather. R. C. BOWKER and E. L. WALLACE (J. Amer. Leather Chem. Assoc., 1928, **23**, 82—91).—A preliminary report describing the preparation of samples of different tan-nages and their impregnation with acid solutions.

D. WOODROFFE.

Clarification of waste tannery liquors and their utilisation for agricultural purposes. V. HLAVINKA (Sbornik Ceskoslovenké Akad. Zemědělské, 1927, **2**, 41—54; Chem. Zentr., 1927, II, 967).—The inorganic constituents, with the exception of compounds of chromium and arsenic, of tannery waste liquors are usually harmless, and no danger is likely to arise from bacteria unless pathogenic bacteria are present; these are only imperfectly removed by treatment with chlorine or bleaching powder. To obtain efficient sedimentation the tanning liquors should be kept separate from the lime-pit liquors. Sedimentation is carried out

in sand pits and in raking plants where the chemical reactions are allowed to complete themselves so that coagulation of suspended matter is accelerated. The slime obtained contains 98% of the inorganic and 75% of the organic solids suspended in the liquors. The clarified liquors retain only 10% of the chromium content, whilst the slime has a nitrogen content of 5—6%. After drying to a water content of 60% it may be used as a fertiliser.

A. R. POWELL.

Determination of residual nitrogen for the evaluation of gelatin products. T. VON FELLEBERG (Z. Unters. Lebensm., 1927, **54**, 481—483).—Bacterial action causes decomposition of proteins resulting in an increase of the non-precipitable nitrogen. The residual soluble nitrogen after precipitation with phosphotungstic acid was determined and calculated as a percentage on the total nitrogen. Good gelatin gave 0.55%, second quality gelatin 1.19%, and glues 1.56—6.53%. After being kept for 19 days at room temperature in a 1% aqueous solution, good gelatin gave 1.9%.

J. R. NICHOLLS.

A new jelly strength tester and some experiments with gelatin gels. A. F. TRACEY (J.S.C.I., 1928, **47**, 94—96 r).—The apparatus described measures the load on a plunger required to break the jelly surface and also gives the depression of the jelly surface under load. The jelly strength is shown to be a surface effect and also to have a temperature coefficient which reaches a minimum about 20°.

PATENTS.

Treatment [curing] of hides and skins. H. DODGE (U.S.P. 1,663,401, 20.3.28. Appl., 8.7.27).—A mixture containing sodium chloride and hydrogen carbonate, potassium nitrate, and formaldehyde is used.

F. G. CROSSE.

Substitute for catgut for use in tennis rackets etc. JOHNSON & JOHNSON (GT. BRITAIN), LTD. From JOHNSON AND JOHNSON (B.P. 285,752, 9.9.27).—Silk strands are deprived of sericin, impregnated with glue (25—30 pts.) and a softening agent, e.g., glycerin (10—15 pts.), with or without a lubricant, e.g., castor oil (20 pts.) or lard (20—30 pts.), and water. The product may be waterproofed by exposure to aldehyde vapour.

D. WOODROFFE.

Preparation of a protective colloid. F. HEIDELBERG (U.S.P. 1,658,289, 7.2.28. Appl., 31.1.21).—A solution of casein, prepared by dissolving 500 g. of casein in water with the addition of 100 c.c. of 20% sodium hydroxide solution at 40°, is fermented with 50 g. of papain, and the precipitate of digested protein is separated, washed, and redissolved in sufficient alkali to form a neutral solution.

F. R. ENNOS.

Automatic scouring machine for treating hides. TURNER TANNING MACHINERY Co., LTD. (B.P. 262,451, 3.12.26. Fr., 3.12.25).

Porous insulating materials (B.P. 265,968).—See IX.

XVI.—AGRICULTURE.

Relation between plant nutrition, plant growth, and the Mitscherlich method for determining the fertiliser requirement of soils. [O. LEMMERMANN,

P. HASSE, and W. JESSEN (Z. Pflanz. Düng., 1928, 7B, 49—98).—In the Mitscherlich method for determining fertiliser requirements of soils, the "factors" are shown to be dependent on environmental conditions and previous manuring. The nitrogen and phosphate factors decrease as other soil conditions improve and as the optimum yield increases. In pot experiments the factors obtained are usually smaller than those by field trials. This is ascribed to improved water conditions maintained in the former. Variations in the factors should be considered in conjunction with "limiting values" and optimum yields. Owing to the inconstancy of the factors, the Mitscherlich method does not give accurate information as to the assimilable food-stuffs present in soils or as to fertiliser needs, although information of considerable value in practice is obtainable.

A. G. POLLARD.

Acidity of moorland soils. B. TACKE, P. ARND, W. SIEMERS, and J. SAFFRON (Landw. Jahrb., 1927, 65, 66—103; Bied. Zentr., 1928, 57, 7—8).—By means of the Tacke-Süchting method it is shown that little or no exchange acidity exists in moor soils. From a consideration of the degree of ionisation of neutral salt solutions and of the free acids, it is concluded that actual neutral salt decomposition occurs in rich organic soils, with the formation of free acids. Liming of moor soils effects a 50% neutral salt decomposition. Sphagnum peat is shown by percolation experiments to bring about a similar decomposition of neutral salts. The ease with which neutral salts are decomposed in humus soils is influenced by the nature of both ions. Sulphates are more easily affected than nitrates, and these in turn more easily than chlorides (cf. B., 1926, 1024).

A. G. POLLARD.

Hygroscopicity and the chemical properties of soils. F. GIESECKE (Chem. Erde, 1927, 3, 98—136; Bied. Zentr., 1928, 57, 4—6).—The hygroscopicity of soil may be represented as the sum of the hygroscopic values of the various mechanical (Atterberg) fractions. Mean values for the individual fractions measured on samples dried at 105—110° are recorded. Results obtained by this process agree with those yielded by the customary direct methods. There is a close parallelism between the hygroscopicity of soils and their contents of iron and aluminium oxides. This relationship is influenced by the ratio of $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3 : \text{SiO}_2$, higher ratios corresponding to decreased hygroscopicity.

A. G. POLLARD.

Slaking of dry soils with water. K. T. HARTLEY (J. Agric. Sci., 1928, 18, 41—45).—A method is described for determining the time required for complete disintegration by water of spheres of dry soil. There appears to be an equilibrium point between colloidal content and pore space at which the time of slaking is at a maximum. Determination of the time of slaking is not, however, a reliable means of comparing soils of different types.

C. T. GIMINGHAM.

Scientific use of nitrogen fertilisers. F. E. BEAR (Ind. Eng. Chem., 1928, 20, 65—70).—A discussion of the modifications in agricultural practice likely to follow on the introduction of cheap synthetic nitrogen in the various forms. Conditions governing the choice

between them are reviewed. It is claimed that under many circumstances "green manuring" in place of the use of fertiliser nitrogen has ceased to be economical, and attention is called to experiments on the use of nitrogen on pastures.

C. IRWIN.

Loss involved by igniting soil fractions during mechanical analysis of soils. F. J. MARTIN (J. Agric. Sci., 1928, 18, 123—130).—With many tropical soils, particularly lateritic soils, the greater part of the loss of weight on ignition of the fractions is due to combined water, not to organic matter. In the clay fraction, the greater the ratio silica:alumina, the less the amount of combined water. Lateritic material is not necessarily evenly distributed among the fractions, and, with these soils, it is considered best to express the results of mechanical analysis on an oven-dry basis, noting that the clay fraction is weighted with the organic matter.

C. T. GIMINGHAM.

Mechanical analysis of tropical soils. J. CHARLTON (Bull. Agric. Res. Inst., Pusa, No. 172, 1927, 9 pp.).—In order completely to disperse the clay in soil suspensions, it is necessary to shake the sample with alkali in an end-over-end shaker for at least 24 hrs. at a temperature of not less than 25°. Pretreatment with hydrogen peroxide does not obviate the necessity for the above prolonged shaking. Ammonia solutions of greater strength than is customary do not hasten the dispersion. "Hyperol" (a solid compound of urea and hydrogen peroxide) is more efficient than ordinary hydrogen peroxide solutions in aiding the dispersion of clay in humus soils. Hydrogen peroxide solutions frequently contain sufficient phosphoric acid to cause appreciable losses of silica and iron sesquioxide from soil suspensions.

A. G. POLLARD.

Aphiscidal properties of tobacco dust. H. C. HUCKETT (New York Agric. Exp. Sta. Tech. Bull., 1926, No. 121, 1—29).—The toxicity of tobacco dust is dependent on the nicotine content and on the period of aphid exposure. The toxicity is increased by the addition of calcium hydroxide (1:1), particularly when the nicotine content is low. Tobacco dusts so diluted are as effective as pure tobacco dust.

CHEMICAL ABSTRACTS.

Effect of iodine manuring on the iodine content of plants. J. B. ORR, F. C. KELLY, and G. L. STUART (J. Agric. Sci., 1928, 18, 159—161).—The results of experiments with oats, mustard, peas, and other plants, in water culture and in soil, show that addition of potassium iodide affords a simple means of increasing the iodine content of food plants.

C. T. GIMINGHAM.

Technique of seed testing. ANON. (Landw. Versuchs-Stat., 1927, 107, 1—64).—Details are recorded of sampling methods and of methods of testing germinating power, vigour, purity (of species and strain), weight and volume, husk and milling value (of cereals).

A. G. POLLARD.

Production of ammonium phosphates. MÜCKENBERGER.—See VII. **Tannery liquors in agriculture.** HLAVINKA.—See XV.

PATENTS.

Dehydration of vegetable substances or products of organic origin. B. J. OWEN (B.P. 285,115, 5.11.26).—Vegetable matter (e.g., sugar-beet slices) is dried in thin

layers by means of heated air. Temperature, pressure, and rate of air current are so controlled that the mass of vegetable matter does not become overheated.

A. G. POLLARD.

Herbicide. G. E. BENSEL (U.S.P. 1,657,062, 24.1.28. Appl., 28.12.25).—Arsenic trioxide is heated with hydrochloric acid to give a solution of arsenic trichloride which is mixed with ferrous sulphate and acid sludge from the treatment of petroleum.

T. S. WHEELER.

Phosphorus material. H. W. CHARLTON, Assr. to AMER. CYANAMID CO. (U.S.P. 1,662,489, 13.3.28. Appl., 2.8.23).—Phosphoric anhydride is mixed with calcium cyanamide.

H. ROYAL-DAWSON.

[Mechanical spray]-treatment of growing plants etc. J. C. SAVAGE (B.P. 285,515, 10.8.26).

XVII.—SUGARS; STARCHES; GUMS.

Treatment of beet-sugar factory effluents. O. SPENGLER (Trans. Inst. Chem. Eng., Advance Proofs, March, 1928, 25—30).—The most important of the recent developments in the methods of dealing with beet-sugar factory effluents are the Hirschfelder system of clarifying the conveying and washing water, and the double fermentation or the fermentation and putrefaction processes for the disposal of the pulp press and diffusion waste-water. In the Hirschfelder system a number of basins are arranged round a sludge pond. The raw effluent passes through the basins in succession, the sludge being deposited therein until the first basin, which receives the largest deposit, is filled; the effluent is then discharged into the next basin and those following until in turn the next basin is filled. As each basin is filled the sludge in it is discharged into the central pond, whence it is pumped into the sludge field. In the Hildesheim double fermentation process the hot effluents from the battery and the pulp presses are de-pulped and then led into the first settling pond in which auto-fermentation takes place. The water thence overflows into a second pond where sufficient lime is added to neutralise 80—90% of the acid. The lime sludge settles and a second fermentation takes place. After passing through other ponds the water is conveyed to irrigation fields, in which the acids still in the water are neutralised by the lime used for fertilisation, and the albuminous substances are precipitated. By this process it is possible to purify sugar factory effluents so well that they may be discharged without objection even into small rivers.

A. B. MANNING.

Sugars in tanning materials. CLARKE and FREY.—See XV. Alcohol from Nipah palm. DENNETT.—See XVIII. Starch in hazel nuts. GRIEBEL.—See XIX.

PATENTS.

Manufacture of dextrose from starch. W. B. NEWKIRK, Assr. to INTERNAT. PATENTS DEVELOPMENT Co. (U.S.P. 1,661,298, 6.3.28. Appl., 25.4.24. Renewed 7.12.27).—See B.P. 232,938; B., 1925, 967.

Manufacture of starch products. E. STERN (U.S.P. 1,661,201, 6.3.28. Appl., 20.3.26. Ger., 5.8.24).—See B.P. 272,274; B., 1927, 612.

XVIII.—FERMENTATION INDUSTRIES.

Modern malt kilns. A. FERNBACH (J. Inst. Brew., 1928, 34, 119—127).—The difficulties of regulating the heating of a malt kiln with two or three floors have resulted in the development of the Winckler kiln, in which hot air from the furnace is forced by a fan through the green malt which is loaded to a depth of from 3 to 5 ft. The resulting rapid evaporation of the moisture keeps the temperature of the drying malt sufficiently low, whilst the large increase in the saturation point of the air at the high temperature also effects a very rapid drying. The drying and curing processes occupy only about 10 hrs. and no turning is necessary. C. RANKEN.

Malting barleys of 1927. J. STEWART (J. Inst. Brew., 1928, 34, 128—143).—The past season's English and foreign malting barleys are reviewed, and attention is directed to the further serious decrease in acreage in the United Kingdom, which constituted a record. The stocks in California were also the lowest on record. The finest qualities of home barleys came from the Eastern counties, but owing to adverse conditions the crop was all more or less weathered. The corresponding malts from the analytical point of view compared favourably with those of the previous year, with the exception that the soluble nitrogen was low, and there was a greater variation in diastase. Czechoslovakia had the best of the foreign barleys suited for pale ales.

C. RANKEN.

Composition and estimation of barley proteins. L. R. BISHOP (J. Inst. Brew., 1928, 34, 101—118).—A method is developed and standardised for the determination of the proteins in barley. Albumin, globulin, and degradation products are first extracted from the barley by salt solution. Hordein is extracted from the residue by hot alcohol and the nitrogen of the final residue is considered to represent glutelin. Direct determinations of glutelin confirm the results obtained with this method of difference. In samples of barley grown on different soils, and with different manuring, the total nitrogen varied from 1.2 to 2.3%. The percentages of glutelin remained constant at 36% of the total nitrogen. The percentage of hordein increased as the total nitrogen increased, rising from 26 to 40% of the total nitrogen, whilst the percentage of the salt-soluble nitrogen correspondingly decreased from 36 to 24%. External conditions alter the total amount of nitrogen in the grain, but appear to have no influence on the proportions of the proteins in the mature barley. Accordingly, varying "quality" of barleys of equal nitrogen content is not due to variations in the amounts of the individual proteins. It is indicated that hordein is the chief protein which is attacked during malting, and breaks down to give salt-soluble compounds. C. RANKEN.

Citric acid content of grape must and wine. A. HEIDUSCHKA and C. PYRIKI (Z. Unters. Lebensm., 1927, 54, 466—473).—The method used was von der Heide's modification (separation of citric acid from sugars etc. by precipitation with barium chloride in ammoniacal solution and the addition of alcohol to 15% by vol.) of Stalire's process (oxidation of citric acid to acetone-dicarboxylic acid and the latter to the difficultly soluble

pentabromoacetone). 1926 Saxony must contained from 0.126–0.221 g. of citric acid per litre. The corresponding wines showed sometimes more and sometimes less; some contained traces or none. A pure culture of wine yeast was found to have no influence on the change in citric acid, which is assumed to be due to another organism. In 15 foreign wines from different countries the citric acid varied from a trace to 0.267 g. per litre. J. R. NICHOLLS.

Fruit-residue spirits with abnormal odour and taste. T. VON FELLEBERG (Z. Unters. Lebensm., 1927, 54, 483–484).—On three different occasions at wide intervals spirits made from apple marc were found to be contaminated with mustard oil. The abnormality was traced to the drainings from the marc of fallen, un-matured apples, and appeared to have been produced by the fermentation of a substance which may occur in unripe apples, but which disappears before maturity. Unripe apples are therefore unsuitable for the production of potable spirits. On another occasion a spirit was produced with a taste and odour of cherries and in which hydrocyanic acid and benzaldehyde could be detected. Further extractions up to twelve pressings produced similar spirits. It was found that excessive pressing of the marc to obtain as much juice as possible had crushed many apple pips, and these had given the unusual flavour. J. R. NICHOLLS.

Nipah palm as a source of alcohol. J. H. DENNETT (Malay. Agric. J., 1927, 15, 420–432; cf. B., 1927, 263).—Juice obtained by tapping the Nipah palm contains cane sugar but no reducing sugars or alcohol. The tapping process is discussed, together with the transport of the juice to the factory. The average yield of alcohol is 10% (by vol.) of the juice and 1300 gals. of alcohol per acre per annum are estimated to be obtainable. Examination of the soil under Nipah palm shows a 40% decrease in the salt content of the top soil since 1924. Little change occurs in the sub-soil. A. G. POLLARD.

PATENTS.

Manufacture of powdered extract of malted grains. A. W. BOSWORTH, ASSR. to DOUTHITT ENGINEERING Co. (U.S.P. 1,658,027, 7.2.28. Appl., 7.2.25).—The grain, after malting, is dried, mixed with water free from carbonates of sodium, potassium, or ammonium, and subjected to diastatic fermentation at 60° to convert the starch into maltose and dextrin. The aqueous extract is drawn off from the grain, which is washed with water at 60°, and the combined unfermented liquor is diluted with water until it contains 10–15% of solids, and is desiccated by injecting it in the form of a finely-divided spray into a volume of heated air, whereby a non-caramelised powder is produced. F. R. ENNOS.

Manufacture of dihydroxyacetone. I. G. FARBER-ENIND. A.-G. (B.P. 269,950, 26.4.27. Ger., 26.4.26).—A decoction of vegetable materials, such as straw, wood, or hay, is added to glycerin and the mixture fermented by *B. xylinum* or *Acetobakter suboxydans*. The conversion of the glycerin into dihydroxyacetone is concluded in four or five days and the process is not impeded by frothing. C. RANKEN.

XIX.—FOODS.

Examination of the biological properties of milk. M. A. DYCHNO and O. M. BRISKIN (Z. Unters. Lebensm., 1927, 54, 438–446).—Peroxydase in milk is detected by the addition of hydrogen peroxide, potassium iodide, and starch. Raw milk gives a blue colour, milk heated above 60° a brownish-green, and heated above 80° no colour. Catalase is measured by the volume of oxygen liberated in 2 hrs. after the addition of hydrogen peroxide. The average volume of gas liberated from 100 c.c. of milk was found to be 31.9 c.c. from raw milk, 14.5 c.c. from baked (i.e., heated for some time below 100°) milk, and 2.5 c.c. from boiled milk. Reductase is measured by the time of decoloration of methylene-blue, and is correlated with the bacterial content. The fluctuation found was between 10 min. for a raw milk of high bacterial content and 7½ hrs. for a boiled milk, and the higher the acidity of the milk the quicker the reduction. A table is given showing the kinds of bacteria detected. The fermentation test (which indicates gas-forming bacteria) is regarded as of great significance. The milk is kept in a closed tube for 24 hrs. at 40°. The better the quality of the milk the less change is apparent; a good fresh milk remains liquid for 12 hrs. Four classes in descending order of quality are described: (1) homogeneous compact curd without whey or gas, (2) point-like, mottled curd with whey and gas, (3) streaky curd adhering to the walls of the tube, the interstices filled with whey, and (4) flocculent cheesy curd. The usual methods of heating or boiling milk are condemned, but pasteurising for ½ hr. at 60–70°, whilst slightly altering the physical character of the milk, has little effect on the biological properties and is recommended.

J. R. NICHOLLS.

Determination of lecithin-phosphoric acid in egg liqueurs. J. GROSSFELD (Z. Unters. Lebensm., 1927, 54, 450–462).—The liqueur (5 g.) is mixed with 50 c.c. of absolute alcohol and heated just to boiling; the mixture is filtered through an extraction thimble and the filtrate is used for extracting the insoluble matter in an apparatus in which the hot solvent vapours come directly in contact with the thimble. After 1 hr. the extract is mixed with potassium hydroxide and magnesium acetate, evaporated to dryness, and ashed. The ash is dissolved in acid and the phosphoric acid precipitated with molybdate solution. The weight of the precipitate after ignition multiplied by 0.0395 gives the lecithin-P₂O₅. Alternatively, the precipitated phosphomolybdate is determined by titration.

J. R. NICHOLLS.

Loss of chlorine in ashing [of flour]. W. VON BRUCHHAUSEN (Z. Unters. Lebensm., 1927, 54, 485–487).—Samples of flour, which showed up to 0.4% Cl when an aqueous extract was tested by Volhard's process, gave practically nil after ashing. Tests were carried out by adding salt to starch, lactose, and sucrose, and ashing with and without sodium carbonate. In the absence of the latter over 99% of the chloride was lost, and even in the presence of an excess of carbonate the loss was 13–15%.

J. R. NICHOLLS.

Determination of stone-cells in cacao products. E. ALPERS (Z. Unters. Lebensm., 1927, 54, 462–466).—

The number of recognisable stone-cells in a cocoa powder depends on the grinding, as some of the cells are ruptured. Powdered Accra cacao shell gave the following mean microscopical counts for stone-cell number, *i.e.*, the number of whole cells in 1 mg. of the dry fat-free substance:—once ground 9337, twice 7498, three times 5886, and four times 3290. A number of shells from cacaos of different origin were examined for stone-cell content after once grinding with cacao fat to yield a product similar in fineness to the commercial article. The stone-cell number varied from 9337 for Accra to 3574 for Caracas. To deduce the proportion of shell from the stone-cell number of an unknown cacao product, the largest of these values should be taken to avoid an over-estimation. Mixtures made up with from 1—5% of Accra shell and shell-free nib once ground together gave stone-cell numbers corresponding very closely with those calculated. It is considered that the proportion of shell normally present in cacao nib should not exceed 1%, representing 2.2% on the dry fat-free substance. This would give a stone-cell number of 220 if the round number of 10,000 is taken for shell. Since some products may be less finely ground 300—350 is suggested as the upper limit for the stone-cell number of cacao nib.

J. R. NICHOLLS.

Determination of cacao shell. W. PLÜCKER, A. STEINRUCK, and F. STARCK (*Z. Unters. Lebensm.*, 1927, 54, 488).—The authors claim priority for the method of determining cacao shell by a count of the stone-cells.

J. R. NICHOLLS.

Determination of vaseline oil in coffee. D. MAROTTA and M. E. ALESSANDRINI (*Annali Chim. Appl.*, 1928, 18, 37—39).—Vaseline oil, which, in proportions not exceeding 0.5%, is the only polishing agent for coffee permitted in Italy, may be determined as follows: 100 g. of the roasted berries are extracted with three successive portions of 70—80 c.c. of light petroleum (*b.p.* 60—70°), and the residue left after complete expulsion of the solvent from the filtered extract is treated with about 1 g. of coarsely-ground sodium hydroxide and 10—12 c.c. of about 30% hydrogen peroxide solution in 2 c.c. portions, the flask being cooled if necessary to allay the frothing. When the action ceases, the mass is heated for 1½ hrs. in a reflux apparatus, with frequent shaking, with 50 c.c. of 12% alcoholic potassium hydroxide solution, and is then diluted with 50 c.c. of water and extracted with three quantities of ether, vigorous shaking being avoided. The ether layer, freed from alkali by washing with water, is treated with a little anhydrous sodium sulphate and, after about 1 hr., filtered through a dry filter. The residue of this ethereal extract is heated for 15 min. on a water-bath with 5—10 c.c. of glacial acetic acid, which dissolves the impurities but only traces of the vaseline oil. The liquid, together with the acetic acid washings of the vessel, is transferred to a graduated tube about 1 cm. wide, which is shaken and left in a bath at 60—70° for 15 min. The volume of the collected vaseline oil, read after about 1 hr., is diminished by 10% and the remainder divided by 1.12 to obtain the weight of the oil present. With ground coffee, the

extraction with light petroleum is effected in a Soxhlet extractor and is continued for 4 hrs. T. H. POPE.

Detection of apple juice in preserves of other fruits. C. ESPESO (*Anal. Fis. Quim.*, 1928, 26, 25—32).—Adulteration of jams with apple juice is generally shown by the presence of malic acid, or of excess of malic acid when the fruit, *e.g.*, peaches, normally contains this acid. Muttelet's method of determination of citric and malic acids in jams (*B.*, 1922, 726) was found to be unsatisfactory, and a modified procedure was worked out. After preparation of the solution as described by Muttelet the removal of pectin substances is completed by the addition of 2 vols. of 95% alcohol and filtration after 1 day. The precipitate of barium citrate and malate, obtained as described by Muttelet, is dissolved in the minimal amount of very dilute hydrochloric acid, made up to 100 c.c., and neutralised with sodium hydroxide. Half the volume of 95% alcohol is added and the precipitate of barium citrate collected. Since this precipitate may also contain malate, it is dissolved and reprecipitated in the same way. The combined filtrates are evaporated to 25 c.c. and 75 c.c. of 95% alcohol added, precipitating barium malate. The presence of malic acid is confirmed by Piñerúa's reagent (β -naphthol in sulphuric acid), with which it gives a green fluorescence. In cases of adulteration with residues of cider manufacture the acidity is lower and little malic acid is found. The first precipitate of pectin substances is small, but the second is greater than normal. The following results are recorded for the citric acid and malic acid contents, respectively: Red currant jams, 0.02—1.6%, 0—0.075%; red currant and apple (1:1), 0.809%, 0.257%; peach jam, 0.81%, 0.614%.

R. K. CALLOW.

Detection of yeast in the presence of hydrolysed animal products. H. KRAUT (*Z. Unters. Lebensm.*, 1927, 54, 446—449).—The usual process for the detection of yeast in meat extracts (Micko's) depends finally upon the precipitation of yeast gum with alcohol. In the presence of degraded animal products a precipitate with alcohol may not necessarily be due to yeast. For the certain detection of yeast gum the precipitate is dissolved in acid and 3% sodium hydroxide added. After filtering, the addition of Fehling's solution to the filtrate causes a bright blue precipitate with yeast gum.

J. R. NICHOLLS.

Starch in hazel nuts. C. GRIEBEL (*Z. Unters. Lebensm.*, 1927, 54, 477—481).—Hazel nuts are generally assumed to be essentially starch-free, but some seeds are rich in starch although a fair proportion contain only small quantities. The presence of starch-bearing seed particles in certain nut preparations has been considered indicative of peanut, but without a proper microscopical examination this may be erroneous. The starchy seeds which find application in practice for the replacement of almonds *etc.* are peanuts, hazel nuts, and cashew nuts. These have been examined microscopically and the chief diagnostic characteristics are recorded. It is assumed that the starch in hazel nuts is due to incomplete ripening, but no diminution of starch occurred on storing for 1 year ripe seeds containing a small quantity of starch.

In all the nuts examined the starch was either in the seed coat or cotyledons, but not in both.

J. R. NICHOLLS.

Horse radish and its preparations. K. AMBERGER and K. HEPP (Z. Unters. Lebensm., 1927, 54, 435—438).—The results of analysis of several samples of horse radish of known origin are given. In unpreserved specimens only traces of sulphur dioxide were obtained, and these are assumed to have been derived from the mustard oil.

J. R. NICHOLLS.

Light-sensitivity of dyes. STEIGMANN.—See IV. **Determination of caffeine and theobromine.** OEHLI.—See XX.

PATENTS.

Separation and purification of the three phosphorus-containing bodies derived from the proteids contained in egg yolk. S. and T. POSTERNAK (B.P. 268,806, 31.3.27. Switz., 31.3.26).—The mixture of the sodium salts of the three compounds containing phosphorus, obtained as described in B.P. 268,805 (B., 1928, 138) by extraction of egg yolk with boiling alcohol, are dissolved in water and the β -compound is precipitated by means of alcohol. From the filtrate the α -compound is precipitated by addition of a soluble alkaline-earth salt, after which the γ -compound is obtained by addition of alcohol to the filtrate or by precipitation as a compound with a heavy metal, e.g., lead or copper, which is subsequently decomposed in the usual manner. Alternatively, the α - and β -compounds may be separated from the γ -compound by addition of an alkaline-earth salt to the solution of mixed sodium salts, or by saturating this solution with common salt and acidifying with mineral acid, the mixed precipitate being converted through the heavy-metal salts into the alkali compounds from which the β -compound is precipitated by alcohol. The γ -compound is then obtained from the solution by either of the above processes.

F. R. ENNOS.

Desiccation [of fruit juices]. W. H. DICKERSON, ASSR. to INDUSTRIAL WASTE PRODUCTS CORP. (U.S.P. 1,658,078, 7.2.28. Appl., 2.8.22).—A dry powder is prepared from fruit juices etc. by adding sucrose and subjecting the mixture in the form of minute liquid particles to the action of a drying gas previously heated above the critical temperature of the mixture, i.e., the temperature at which caramelisation and melting occur. The heat content of the gas and the proportion of the latter relative to the amount of substance to be desiccated are so maintained that the temperature of the dried product falls below the critical point, owing to the heat absorbed by evaporation, whilst the moisture content of the gas is adjusted so as to prevent the dried particles from coalescing.

F. R. ENNOS.

Manufacture of high-grade apple juice. E. R. DARLING, ASSR. to HYDRAULIC PRESS MANUF. CO. (U.S.P. 1,659,086, 14.2.28. Appl., 10.11.26).—The pomace is treated with diastase at about 60° to break down the starch cells present and thus facilitate recovery of the juice.

T. S. WHEELER.

Extract of malted grains (U.S.P. 1,658,027).—See XVIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Arsenious oxide in neutral and alkaline solution. W. H. MILLAR (Pharm. J., 1928, 120, 214—215).—In the preparation of Liquor Arsenicalis, B.P. the amount of potassium carbonate used is in excess of that actually required to cause dissolution of the arsenious oxide. 8% of the potassium carbonate reacts to form potassium meta-arsenite and, using this amount in the preparation, dissolution is complete. A 1% neutral, stable solution of arsenious oxide can be prepared by adding potassium carbonate solution to a boiling water suspension containing 1% of arsenious oxide until the latter is dissolved. Potassium carbonate equivalent to 4% of the arsenious oxide is required and the solution is neutralised to methyl-orange with 0.1N-sulphuric acid. Addition of potassium sulphate equivalent to the potassium carbonate used does not effect dissolution.

E. H. SHARPLES.

Determination of benzaldehyde in cherry laurel water. F. MORVILLEZ and (Mlle.) DÉFOSSEZ (J. Pharm. Chim., 1927, [viii], 6, 204—210).—Two methods for the determination of benzaldehyde in cherry laurel water are described. The first is a quick, accurate, "sorting" test, and depends on the absorption of potassium permanganate under definite conditions. The absorption is proportional to the benzaldehyde content and, for normal samples of cherry laurel water, ranges from 22 to 29 c.c. of 0.01N-potassium permanganate per 1 c.c. of water. The method is practically unaffected by the presence of either hydrocyanic acid or nitrobenzene but gives high results if stannous cyanide is present. A more accurate method is to oxidise the aldehyde with chromic acid after hydrolysis of the mandelonitrile. The benzoic acid is extracted with chloroform and determined gravimetrically or volumetrically. Results by these methods are compared with those obtained by precipitation of the aldehyde with "melubrin," and they show satisfactory agreement.

E. H. SHARPLES.

Microchemical determination of caffeine and theobromine in drugs. A. OEHLI (Pharm. Acta Helv., 1927, 2, 155—167; Chem. Zentr., 1927, ii, 1989).—The powdered material (0.1—0.3 g.) is extracted with chloroform in a separating funnel after addition of a few drops of ammonia solution. Chloroform and ammonia are completely removed from the united extracts, the residue is boiled with aqueous magnesia free from soda, and the wax, resin, and colouring matter are removed by filtration. The filtrate is neutralised with 1% acetic acid and treated with 1% permanganate solution, the excess of which is removed with 3% hydrogen peroxide solution containing 1% of acetic acid, filtered, and the colourless filtrate extracted several times with chloroform. After removal of the chloroform by evaporation, the caffeine in the residue is determined (a) volumetrically, by the Kjeldahl-Pregl method, or (b) gravimetrically after purification by vacuum-sublimation in a manner described. For guarana, 1—4% sulphuric acid is used instead of ammonia. For the separation of caffeine and theobromine from maté, the differing solubility in benzene is employed. For the gravimetric micro-determination of the xanthine bases in cocoa, 0.1—0.5 g. is heated with aqueous alcoholic sulphuric

acid, then water is added, and the mixture boiled with magnesia. After filtration, evaporation, extraction with water, neutralisation with 1% acetic acid, and treatment with permanganate and hydrogen peroxide, the filtrate is evaporated to a small volume, and percolated with chloroform in the presence of phenol. The residue after removal of phenol and chloroform is dried in a vacuum and sublimed at 200—210°.

A. A. ELDRIDGE.

Sclerocarya caffra, Sond. M. G. BRANDWYK (Pharm. J., 1928, 120, 172, 192—193, 212).—The bark of *Sclerocarya caffra*, Sond., which has been used in the treatment of malaria, has been examined. It contains no constituents volatile in steam, and on extraction with alcohol it gives 29.3% of a solid extract, which contains about 70% of tannic acid and traces of alkaloids. Glucosides and neutral principles are absent. When injected subcutaneously into a cat, the extract had a severe local effect, but was not toxic.

E. H. SHARPLES.

Constituents of Indian essential oils. XXIII. Essential oil from fruits of *Piper Cubeba*, Linn. B. S. RAO, V. P. SHINTRE, and J. L. SIMONSEN (J.S.C.I., 1928, 47, 92—94 T).—Fruits of *Piper Cubeba* grown in Mysore have been found to yield 7.5% of an essential oil. The main constituents of the oil were shown to be *d*-sabinene, *d*- Δ^4 -carene, 1:4-cineole, *d*-1-methyl-4-isopropyl- Δ^4 -cyclohexen-4-ol, and *l*-cadinene. Attention was directed to the genetic relationship of sabinene, 1:4-cineole, and the alcohol. 1:4-Cineole has not previously been found to occur in nature.

Essential oil from the leaves of *Caesalpinia Sappan*, L. P. VAN ROMBURGH (Proc. K. Akad. Wetensch. Amsterdam, 1927, 30, 840—843).—The hydrocarbon $C_{10}H_{16}$, d^{20}_D 0.813, n^{20}_D 1.473, which is isolated by repeated fractional distillation of the essential oil obtained by steam distillation from the leaves of *Caesalpinia Sappan*, L., yields a nitrosite identical with the nitrosite of *d*- α -phellandrene. By reduction with sodium and boiling alcohol, a hydrocarbon, b.p. 63°/15 mm., d^{20}_D 0.800, n^{20}_D 1.4581, α_D +15.48°, which readily absorbs bromine to yield a product from which a small quantity of dihydro-ocimene tetrabromide, separates. Hydration of the original hydrocarbon with acetic and dilute sulphuric acids yields a viscous alcohol, b.p. 115—122°/30 mm., d^{20}_D 0.890, n^{20}_D 1.484 (liquid urethane). The density of both the hydrocarbon and its reduction product increases on keeping. It is concluded that the hydrocarbon is probably a mixture of *d*- α -phellandrene with an olefinic terpene.

J. W. BAKER.

PATENTS.

Halogeno-tert.-alkyl carbamates. A. W. DOX and L. YODER, Assrs. to PARKE, DAVIS, & Co. (U.S.P. 1,658,231, 7.2.28. Appl., 9.10.22).—A metallic halogeno-tert.-alkoxide is treated with carbonyl chloride in presence of an inert solvent, e.g., benzene, and the resulting chloroformate is treated with ammonia to give the corresponding halogeno-tert.-alkyl carbamate, of therapeutic value as a non-toxic sedative. *gem*.-*Trichloro-tert*.-butyl carbamate m.p. 102°, and *gem*.-*dichloro-tert*.-butyl carbamate, m.p. 122°, are described. T. S. WHEELER.

3':5'-Dinitro-4'-hydroxy-*o*-benzoylbenzoic acid.
3':5'-Diamino-4'-hydroxy-*o*-benzoylbenzoic acid

1:3-Diamino-2-hydroxyanthraquinone. I. GUBELMANN, H. J. WEILAND, and O. STALLMANN, Assrs. to NEWPORT Co. (U.S.P. 1,659,358—1,659,360, 14.2.28. Appl., 29.11.26).—(A) 4'-Hydroxy-*o*-benzoylbenzoic acid is nitrated with a mixture of nitric and sulphuric acids at room temperature to 3':5'-*dinitro-4'-hydroxy-*o*-benzoylbenzoic acid*, m.p. 199°. (B) This compound on reduction yields 3':5'-*diamino-4'-hydroxy-*o*-benzoylbenzoic acid*. (C) The amino-compound (B) is condensed by treatment with concentrated sulphuric acid at 180° to 1:3-*diamino-2-hydroxyanthraquinone*, m.p. 300—305°.

T. S. WHEELER.

Manufacture of tasteless alkaloid preparations. H. HEIMANN (G.P. 444,388, 31.10.24).—Alkaloids are combined with humous substances obtainable from peat, lignite, decayed wood, or other natural sources, or synthetically from sugars, phenols, or proteins; aqueous, alcoholic, or other solutions of the bases or their salts (particularly acetates) may be utilised. The reaction is of the nature of a base-exchange, but differs from the permutit process in that it is the hydrogen ions of the humic acids which take part, liberating acid from the alkaloid salts. The products, washed with pure solvent and then, if desired, with water, are quite tasteless, water-insoluble powders, which can be made up into pastilles with addition of suitable flavouring matters. The alkaloids are regenerated from the products by the action of neutral salts (sodium chloride) or dilute acids, and hence the products have in the organism the same effect as the alkaloids themselves. By shaking an aqueous solution of caffeine with lignite humus for 19 min. at 40°, filtering, and washing with water, 60% of the caffeine is obtained as insoluble humate. Alcoholic cinchonidine with humus from crude sugar, and aqueous quinine hydrochloride with calcium humate behave similarly. C. HOLLINS.

Manufacture of water-soluble double compounds of digitoglucotannoids, the total digitalis glucosides being in their natural form. KNOLL & Co. (G.P. 444,064, 13.9.24).—Digitoglucotannoids are treated with solutions of carbamide, sodium salicylate, or sodium benzoate in water or other solvent (alcohol), giving solutions of double compounds which are used as such or are evaporated to dryness in a vacuum. The neutral solutions may be sterilised. The combination also takes place, on account of the hygroscopic nature of the tannoids, when the dry mixtures are exposed to moist air. C. HOLLINS.

Preparation of heterocyclic arsenic compounds. A. BINZ and C. RÄTH (U.S.P. 1,661,871, 6.3.28. Appl., 2.4.26. Ger., 4.11.25).—See B.P. 250,287; B., 1926, 512.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENTS.

Two-colour kinematograph films. J. E. THORNTON (B.P. 285,262, 8.3.27).—A two-colour film is built up from two thin component films, each consisting of a support and a sensitive colloid layer on each support, one layer being coloured orange-red and the other blue-green before printing, the layers being printed from the back and developed in hot water to produce coloured reliefs,

and the films then being cemented together with their supports in contact. One coloured image is reversed with respect to the other, and the layers are printed through the back of the supports to which they are attached. The films may be cemented together after printing, and the relief images are protected with varnish.

W. CLARK.

Film material for the production of multi-colour transparencies. J. E. THORNTON (B.P. 285,227, 20.1.27).—For producing multi-colour positive transparencies built up by cementing in superposition the gelatin layers with no waterproof celluloid supports, various quadruple-width webs of non-sensitised material are described. A sensitising agent may be absorbed from solution into the colloid film layer after it is spread on the temporary layer, or mixed with the colloid of the film-layer. [Stat. ref. to B.P. 26,828 and 25,084 of 1912, and 26,786 of 1911.]

W. CLARK.

Manufacture of multi-colour kinematograph films. J. E. THORNTON (B.P. 285,228, 28.1.27).—A multi-colour film, consisting only of the gelatin layers with no celluloid supports, comprises four differently-coloured, sensitive, colloid film layers, each layer being printed with a component image of a four-colour picture, and the layers being cemented together in register to form a single film strip of positive pictures. A cellulose cement is used as binding agent. Exposed parts of the colloid film are protected with cellulose varnish. The compound film may also consist of three or two component colloid layers.

W. CLARK.

Production of partial negatives or diapositives for multi-colour printing. A. NEFGEN (U.S.P. 1,661,079, 28.2.28. Appl., 27.2.23. Ger., 7.3.22).—The preparation of the partial plates is carried out from a diapositive of the original which has been coloured or painted with all the basic soluble colours.

W. CLARK.

Production of a colour photograph. L. D. MANNES and L. GODOWSKY, JUN. (U.S.P. 1,659,148, 14.2.28. Appl., 4.8.23. Cf. U.S.P. 1,516,824; B., 1925, 115).—The photographic film carries two superposed emulsions, that next to the support being sensitised to record orange-red sensations, and the other being treated to record blue-green sensations, and, in addition, with a yellow dye to exclude blue and violet rays from the lower emulsion. The exposed negative is developed in metol and bleached in potassium ferricyanide solution, and the upper image is redeveloped in amidol. The lower image is toned blue with ferrous chloride solution, and the upper image is treated with cupric ferricyanide as a mordant, and with fuchsin. The negative is completed by treatment with a yellow basic dye. The positive is formed by exposure of a similar film through the negative, and is developed in the same way.

T. S. WHEELER.

Photographic medium and process. M. C. BEEBE, A. MURRAY, and H. V. HERLINGER, ASSRS. to WADSWORTH WATCH CASE CO. (U.S.P. 1,658,510, 7.2.28. Appl., 15.3.26. Cf. U.S.P. 1,587,269—1,587,274; B., 1926, 773).—A cellulose ester, *e.g.*, cellulose acetate, is sensitised with a suitable organic halogen compound, *e.g.*, iodoform (1%). The image formed on exposure to light

is developed in a silver reducing solution containing silver nitrate and ammonia.

T. S. WHEELER.

Photography. N. SULZBERGER (U.S.P. 1,658,177, 7.2.28. Appl., 4.6.23).—The use of a developer containing phenylhydroxylamine oxalate is claimed to give sepia tones.

T. S. WHEELER.

Screens for photographic purposes, photo-mechanical printing processes, etc. A. R. TRIST (B.P. 286,340, 4.11.26).

XXII.—EXPLOSIVES; MATCHES.

Gases from blasting in tunnels and metal-mine drifts. E. D. GARDNER, S. P. HOWELL, and G. W. JONES (U.S. Bur. Mines, Bull. 287, 1927, 96 pp.).—Analyses have been made of the gases produced by blasting in tunnels and underground mines in order to determine the effect of varying the explosive and the methods of drilling and blasting on the formation of toxic gases. Accurate and representative sampling was difficult in some mines, but in one series of experiments the gases were confined by brattices and were thoroughly mixed before a sample was taken. The results of the sampling indicate that the gases produced by blasting are not always the same as those obtained in the Bichel gauge. Under normal conditions the only toxic gas produced in dangerous quantities is carbon monoxide, but blasts in massive sulphides give rise to dangerous quantities of hydrogen sulphide and sulphur dioxide. Oxides of nitrogen are produced only in harmless amounts even with explosives containing an excess of 3–5% of oxygen. Less carbon monoxide is made by blasting with some gelatin dynamites than with others, and the gases from two commercial "gelatins" improved during the course of the tests. The oxygen balance of the explosive is the main factor in determining the amount of carbon monoxide produced, but its condition has also an effect; gelatin dynamite that is too insensitive to detonation for efficient work should not be used. The manner of drilling does not appear to have any definite effect on the composition of the gases, but in hard rock the use of stemming reduces the amount of carbon monoxide. Removal of the wrapper from the cartridge has the same effect, but also lowers the strength of the explosive. Less carbon monoxide appears to be formed by blasting in wet ground than in dry rock.

A. B. MANNING.

PATENTS.

Production of gelatinised explosives or smokeless powders. DYNAMIT A.-G. VORM. A. NOBEL & CO. (B.P. 282,631, 27.9.27. Ger., 21.12.26).—Gelatinous dynamites or smokeless powders are made by gelatinising with nitrocellulose a solution of one or more nitrates of solid multivalent alcohols in nitroglycerol or nitroglycol. Sorbitol and mannitol are suitable alcohols for this purpose. Diphenylamine may be added as a stabiliser and urea as an aid to gelatinisation.

S. BINNING.

Gelatin dynamite. C. D. PRATT, ASSR. to ATLAS POWDER CO. (U.S.P. 1,658,816, 14.2.28. Appl., 25.3.27).—The keeping properties of gelatin dynamite are improved by addition of dried beet pulp.

T. S. WHEELER.

Explosive priming mixture. A. E. WHITE. From REMINGTON ARMS Co. (B.P. 285,232, 24.1.27).—A non-corrosive priming mixture for ammunition contains mercury fulminate as initial explosive, an oxidiser containing a salt of an alkaline-earth metal, either alone or with another oxidising salt of a heavy metal, and a reducing agent such as a thiocyanate of a heavy metal, alone or mixed with another reducing agent. Barium nitrate with or without lead nitrate may be used as the oxidising agent and antimony sulphide as the second reducing agent. S. BINNING.

Explosive composition. W. O. SNELLING and G. A. RUPP, Assrs. to TROJAN POWDER Co. (U.S.P. 1,659,449, 14.2.28. Appl., 17.7.26).—The addition of 1–5% of ferrosilicon to explosives containing nitrostarch is claimed. T. S. WHEELER.

XXIII.—SANITATION; WATER PURIFICATION.

Intensive bacterial oxidation [of activated sludge]. Oxidation of ammonia to nitric acid. I–IV. G. J. FOWLER, Y. N. KOTWAL, R. V. NORRIS, S. RANGANATHAN, and M. B. ROY (J. Indian Inst. Sci., 1927, 10A, 97–116).—The conditions under which intensive nitrification can proceed, using the activated sludge process, have been studied. It is shown that the sludge must be acclimatised to the peculiar conditions by increasing the strength of the ammonia solution slowly as the capacity to deal with it increases, the theoretical quantity of calcium carbonate to neutralise the acid produced being added on each occasion. It was found that the maximum amount of ammoniacal nitrogen which could be oxidised under these conditions was 0.25 g./litre per day. Increasing concentrations of calcium nitrate were found to reduce progressively the rate of reaction, the maximum concentration obtainable being 7.5% when nutrient salts also were added and 5.56% when such salts were absent. The influence on the reaction of various salts was investigated, of which ferrous sulphate was found to have a beneficial effect. The optimum p_H value is shown to be between 7.0 and 8.0. C. JEPSON.

Technique and significance of the biochemical oxygen demand determination [of water]. F. W. MOHLMAN, G. P. EDWARDS, and G. SWOPE (Ind. Eng. Chem., 1928, 20, 242–246).—The various methods of carrying out the test and comparative results obtained are discussed. A description is given of the method adopted in the Sanitary District of Chicago which includes the use of a standard water consisting of distilled water and 500 p.p.m. of sodium bicarbonate. The rate of satisfaction of biological oxygen demand in sewage and polluted streams is shown to follow a two-stage curve, the first, which is satisfied in 5–12 days, representing a carbonaceous oxidation, and the second, conversion of nitrogenous material into nitrate. C. JEPSON.

Method for determining the biochemical oxygen demand [of water]. F. SIERP (Ind. Eng. Chem., 1928, 20, 247).—An apparatus is described by means of which the determination is made by direct absorption of gaseous oxygen by the undiluted sample. It is possible to read off the oxygen demand when desired, and

many errors unavoidably incurred in other methods are eliminated. C. JEPSON.

Cyanide-citrate pour-plate medium for direct determination of the colon-aërogenes content of water and sewage. R. E. NOBLE (J. Amer. Water Works' Assoc., 1928, 19, 182–192).—The medium described is a modified Ayers-Rupp medium containing two new ingredients, viz., ferric citrate and potassium ferrocyanide. It is claimed that with this medium the time required to obtain the *B. coli* index number may be reduced by 40–50%, and the results obtained are more correct than with the lactose broth tube-method in general use. It also promises to afford a ready means of distinguishing between organisms of faecal and non-faecal origin. C. JEPSON.

Colorimetric p_H determinations [of water] in a neutral atmosphere. H. F. MUER and F. E. HALE (J. Amer. Water Works' Assoc., 1928, 19, 201–206).—The effect of the laboratory atmosphere on colorimetric p_H determinations of water may be considerable. It is suggested that the test should be carried out in stoppered bottles previously filled with air neutralised by passing through potassium hydroxide solution and lime water. C. JEPSON.

Monochloroamine as a disinfectant for swimming baths. W. OLSZEWSKI (Chem.-Ztg., 1928, 52, 141–142).—Chlorine, hypochlorous acid, and chloramine form a series of bactericidal agents of which the efficiency increases in that order. The most satisfactory method for the preparation of monochloroamine is by the action of ammonia on free hypochlorous acid. L. M. CLARK.

Concrete control methods in the construction of a filtered-water reservoir. W. C. MABEE (J. Amer. Water Works' Assoc., 1928, 19, 193–200).—The ratio of water to cement when mixing concrete was made part of the contract when constructing a covered, filtered-water reservoir for the Indianapolis Water Company. A 28-day strength of not less than 2500 lb./in.² in compression was aimed at and achieved by careful control of this ratio, and by study of the conditions relative to aggregates, time of mixing, distribution, placing, puddling, etc. On test, under 14-ft. head, the tank lost only 0.3% of its volume in 24 hrs. C. JEPSON.

PATENTS.

Purification of sewage and analogous liquids. A. V. DE LAPORTE, F. A. DALLYN, and F. R. MANUEL (U.S.P. 1,658,974, 14.2.28. Appl., 12.4.26).—The sewage is agitated in presence of oxygen and chlorine with a mixture of aluminium and iron salts to precipitate and oxidise colloidal matter. T. S. WHEELER.

Manufacture of base-exchange substances for the removal of iron and manganese compounds and hardness from drinking and boiler-feed waters. W. GRADENWITZ (G.P. 445,377–8, [A] 14.9.18, [B] 19.9.18. Addn. to G.P. 403,263; cf. U.S.P. 1,493,455; B., 1924, 691).—(A) Burnt clay or brick is subjected to a short treatment with concentrated hydrochloric acid; the product is then sufficiently active as a base-exchanging material without further calcination, or (B) it may subsequently be treated with ammoniacal calcium chloride solution before use. A. R. POWELL.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAY 11, 1928.

I.—GENERAL; PLANT; MACHINERY.

Porosity and permeability as characteristics of porous bodies. O. PHILIPP (Chem. Fabr., 1928, 152—153; cf. Howe and Hudson, B., 1928, 39).—An apparatus is described for determination of permeability by measuring the volume of air which passes through per m.² per min. at a pressure of 25 mm. The results are compared for porous plates of the same thickness prepared by binding particles of definite sizes with varying proportions of binder. S. I. LEVY.

Filter system [for the removal of small quantities of fine solids from large volumes of liquid]. K. KELLER (Chem.-Ztg., 1928, 52, 111—113).—The filter consists of a large closed chamber of sheet metal containing a number of thin vertical filter elements, the insides of which are connected with a receiving tank for clarified liquid. The elements consist of stout metal frameworks on both sides of which are stretched sheets of fine wire gauze. To start the filter a volume of liquid equal to the capacity of the chamber is stirred vigorously in the feed tank with a quantity of fibrous material, such as asbestos, equivalent to 150 g./m.² of filter surface; this mixture is then pumped into the filter chamber under a pressure of 5 m. of water, whereby a thin film of filtering material is formed over the wire gauze surfaces of the filter elements. After this film has formed, filtration may be carried on continuously, the liquid being passed to the filter from a feed tank situated 2—3 m. above it. After operations have ceased, the cake of solids may be readily removed in one piece from the surface of the gauze. A. R. POWELL.

Properties of powders. IX. Scattering of light by graded particles in suspension. T. M. LOWRY and M. C. MARSH (Trans. Faraday Soc., 1928, 24, 195—201).—See B., 1928, 175.

PATENTS.

Regenerative reversing furnace. J. KENNEDY (U.S.P. 1,657,401, 24.1.28. Appl., 7.5.21).—During the first half of each regenerative cycle the outgoing hot gases are passed through the entire space of one regenerator while the incoming air is passed first through one portion and thereafter through another portion of the other regenerator, and during the second half-cycle similar operation is effected with the flow through the furnace reversed and the functions of the regenerators interchanged. H. HOLMES.

Furnaces or gas-producers burning pulverised solid or liquid fuel. E. S. SUFFERN, GASIFIED FUEL, LTD., H. E. HAZELHURST, and O. MARGETSON (B.P. 284,738, 1.9.26 and 10.2.27).—The fuel is incompletely burnt in a primary combustion chamber, into which the

fuel is projected at an angle, and is deflected from the walls so that the path of the fuel and primary air forms a loop; the outlet to the secondary combustion or heating chamber is restricted so that the temperature of the primary chamber will be maintained and the flame passing through the construction not extinguished. The primary chamber may be portable and applied to the front of a Lancashire or similar boiler.

B. M. VENABLES.

Combined drying-chamber plant and grate furnace. G. HERRMANN (B.P. 285,546, 15.11.26).—A firing chamber, heat-absorbing checker-work chamber, a drying chamber, and a chimney are arranged adjacent to each other. The checker-work is first heated by the fire, the gases not passing through the drying chamber. After the fire is extinguished air is drawn through all three chambers.

B. M. VENABLES.

Drying channel or tunnel. H. WINKLER (B.P. 284,747, 28.10.26).—A drying channel is divided into various zones with re-heating elements alongside. A portion of the air in each zone circulates continually through the drying channel and heater of that zone, but a portion is taken from the pressure side of the fans or wind wheels and delivered to the next heating zone.

B. M. VENABLES.

Conical crushing mill. C. F. SMITH, A. C. THIELE, and L. P. WALKER, ASSTS. to SMITH ENGINEERING WORKS (U.S.P. 1,663,355, 20.3.28. Appl., 21.5.26).—The mill comprises a rotating conical casing with a conical crusher inside it mounted on a shaft set at an angle to the axis of the casing. The supports for the shaft are situated beyond the ends of the casing, one acting as a pivot and the other constructed so as to allow a certain freedom of movement of the shaft and crusher. L. A. COLES.

Vibrating apparatus for screening, sorting, drying, etc. A. KUHN (B.P. 285,638, 2.2.27).—A W-shaped spring for supporting the vibrating frame in flexible relation to the fixed support is described. The supporting rod for the frame is secured to the centre points of the W-springs. Springs of other shape (e.g., helical) may be used. B. M. VENABLES.

Separating or classifying machine. W. A. RIDDELL (U.S.P. 1,661,501, 6.3.28. Appl., 10.12.25).—A hopper is provided with a number of longitudinal rotating rollers, the distance between which is capable of adjustment, together with a number of longitudinal guide plates held firmly above the gaps between the rollers. A. R. POWELL.

Treatment of granular material in mixing and filtering vats. J. CARLIER (B.P. 285,979, 24.11.26).—Fluid under pressure is supplied to the mass through a

hollow rake which is gradually lowered into the vat while rotating. B. M. VENABLES.

Centrifuges. L. ALTPETER (B.P. 284,831—2, 24.12.26).—(A) In a centrifuge of which the bowl comprises two cones base to base which are separated to discharge collected solids, a method of sealing the joint is described in which the packing material is the collected solid itself. (n) In a centrifugal bowl, methods are described by which clear, separated liquid may be drawn off through outlets which are part of the bowl, but which may be adjusted to different depths while the bowl is running. B. M. VENABLES.

Centrifugal separators. LAUGHLIN FILTER CORP., Assees. of W. C. LAUGHLIN (B.P. [A] 279,896, 29.10.27. U.S., 30.10.26. [E] 283,918, 8.11.27. U.S., 20.1.27).—The material is fed through a hollow shaft (which may be horizontal) to the annular and triangular space within a frusto-conical rotor, and thence through ports to the exterior thereof. On the exterior is a helical blade or upstanding flange which is in close contact with an outer frusto-conical rotor. The two rotors are driven positively at slightly different speeds (the outer one being on a sleeve shaft), so that the heavy collected material is pushed by the helix to the small end of the outer cone and there discharged. The lighter material is discharged round an annular baffle extending inwards at the base of the outer cone. In (n) holes are provided at or near the root of the helix at the smaller end, so that semi-separated material has a chance to flow back and become properly separated. B. M. VENABLES.

Centrifugal drying apparatus. E. D'HOOGHE (B.P. 283,466, 9.1.28. Belg., 8.1.27).—The whole machine is slung by ball and socket bolts so as to be quite free from rigid restraint. The drive is by friction bevels, which are moved out of engagement for stopping, and a band brake is provided; both are operated by the same lever. B. M. VENABLES.

Washing of minerals by means of liquid streams. A. FRANCE (B.P. 285,987, 24.11.26).—The pulp flows in a launder of inclination suitable to produce stratification, and the heavier mineral drops through slots in the bottom of the launder against a rising stream of water, and is re-treated by other rising streams of water in a separate apparatus. B. M. VENABLES.

Controlling the movement of masses of solids of various sizes. L. E. W. PLODA (U.S.P. 1,663,173, 20.3.28. Appl., 26.12.23).—Air entrapped by solids of unequal size in their movement towards an outlet at the bottom of a container is released and passed to vents before the solids reach the outlet. F. G. CLARKE.

Making and delivering quantitative mixtures. T. MACRAE (U.S.P. 1,663,206, 20.3.28. Appl., 25.6.23).—Predetermined charges of materials, after separation from bulk, are spread in the form of thin parallel sheets and mixed by forcibly impacting the sheets against each other. The mixture is separated into charges which are successively mixed with water in a chamber and discharged at the place of use under pressure. F. G. CLARKE.

Emulsification apparatus. G. C. HURRELL (B.P. 285,159, 13.11.26).—The emulsifier, suitable for manufacture in small sizes, comprises a piston pump with an

automatic inlet valve, but no outlet in the ordinary sense, the emulsion being forced through the working clearance between the cylinder and piston. B. M. VENABLES.

Emulsifying machine. A. C. E. ANDERSEN-ORRIS (B.P. 284,910, 17.5.27).—The materials are passed through small circuitous passages formed between a rapidly-rotating disc and a plate screwed thereto. B. M. VENABLES.

Automatic hydraulically-operated extraction presses. SOC. ANON. DES ATELIERS ARMAND & DÉOUNE (B.P. 262,072, 1.11.26. Fr., 26.11.25).—The material is pressed between a hydraulic ram and a fabric-covered end bed-plate, being surrounded by a sliding collar. The ram being withdrawn, the material is charged by gravity through an aperture in the collar; during the first motion of the ram this aperture is closed, the ram continues and expresses the liquid, the ram is slowly withdrawn, and meanwhile the collar is moved in the same direction at a faster speed by means of auxiliary rams. The pressed cake is thus freed and can be withdrawn either upwards or downwards by moving the filter cloth, which is in the form of a long strip wound on rollers at each end. The collar and, if desired, the face of the ram are lined with filter fabric. B. M. VENABLES.

[Edge] filters. E. W. PATTISON. From METAL EDGE FILTER CORP. (B.P. 285,127, 9.11.26).—In an edge filter of which the pack is composed of metal sheets or the like, spacing of the sheets is effected by electro-deposition of metal on them locally, the spots being formed by coating the rest of the surface with insulating material which is removed after the electrodeposition. B. M. VENABLES.

Filtering apparatus and filters for gasoline or other liquid fuels. SOC. DU CARBURATEUR ZÉNITH (B.P. 279,382, 30.5.27. Fr., 23.10.26).—A float-operated valve is provided to discharge air or gases from the prefilter to the filtrate side, in cases where the pressure available across the filter is insufficient to force the gas through the filter. B. M. VENABLES.

Carrying out catalytic chemical processes. J. TRAUTMANN (B.P. 261,787, 20.11.26. Ger., 21.11.25).—In carrying out chemical reactions in which a molten metal, alloy, or salt in a finely-divided form is used as a catalyst, a bath of the molten catalyst is formed in the reaction chamber and subjected to a centrifugal or threshing operation so as to break up the catalyst into fine spherical particles which are brought into contact with the reacting materials. Means are provided for heating the charge electrically with either external or internal heating elements. A. R. POWELL.

Catalytic apparatus. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,660,511, 28.2.28. Appl., 19.1.27).—An apparatus for carrying out catalytic reactions comprises at least one automatic gas-cooled converter with means for providing a cooling effect which increases with the velocity of the reacting gases through the converter, and at least one converter of much less effective cooling capacity, the connexions between the two converters being also provided with means for cooling the gases as they pass from one to the other. The first converter contains a layer of catalyst in which are embedded heat-exchanging elements, and means are

provided for passing the reacting gases through these elements out of direct heat-exchange with the catalyst and back through part of the elements in direct heat-exchange with the catalyst, thence through the catalyst.

A. R. POWELL.

Production of cold. K. BERGL and W. DIETRICH (B.P. 268,358, 23.3.27. Ger., 29.3.26).—An inexpensive organic fluid, which is soluble in water and which will evaporate at about 0° in a moderate vacuum, is placed in a closed container surrounded by the material to be chilled and evaporated by means of a water-jet pump. The liquid is preferably soaked up by an absorbent material in the cooling chamber.

B. M. VENABLES.

Manufacture of friction bodies for brake and clutch purposes. KIRCHBACH'SCHE WERKE KIRCHBACH & Co. (B.P. 272,478, 24.5.27. Ger., 9.6.26).—Blocks adapted to minimise overheating are composed of impregnated asbestos fibres mixed with finely-divided grey cast-iron, the metal being preferably in excess. The impregnating material is a bakelite or other artificial resin varnish.

H. HOLMES.

Adsorption treatment of fluids for the purification thereof or separation of valuable constituents therefrom. H. HOLLINGS, S. PEXTON, W. A. VOSS, and GAS LIGHT & COKE Co. (B.P. 284,758, 2.11.26 and 26.1.27).—Adsorbent material, such as the activated charcoal used for extraction of benzol from coal gas, is protected from the deleterious action of iron carbonyl and other "adventitious" impurities by a pre-filter or other means, and formation of iron carbonyl is prevented in the absorbing vessel by coating (or constructing) the walls with iron-free material.

B. M. VENABLES.

Colloidal dispersion of materials in fluids. J. BOURDAIS (B.P. 285,258, 1.3.27).—The material is forced through a fixed or rotating grid-like structure preferably by means of a centrifugal rotor provided with jets.

B. M. VENABLES.

Introduction of semi-liquid materials into high-pressure vessels. SYNTHETIC AMMONIA & NITRATES, LTD., and H. A. HUMPHREY (B.P. 286,324, 27.9.26).—An auxiliary pressure-resisting vessel is filled with the material, which is then forced out of the auxiliary vessel into the main vessel by means of another fluid at high pressure. The two fluids may be in direct contact, or the auxiliary vessel may comprise the smaller cylinder of a two-diameter hydraulic piston operated by the high-pressure fluid.

B. M. VENABLES.

Air classifiers. A. H. STEBBINS (U.S.P. 1,660,682—7, 28.2.28. Appl., [A] 8.1.26, [B, C] 24.9.26, [D] 29.1.27, [E, F] 1.2.27).—Various forms of air separator without inherent moving parts are described; in (A) the heavier particles are separated by their inertia in a straight line, and in (B—F) centrifugally.

B. M. VENABLES.

Production of foam for fire-extinguishing purposes. EXCELSIOR FEUERLÖSCHGERÄTE A.-G., and O. TREICHEL (B.P. 263,812, 23.12.26. Ger., 24.12.25).—A suspension of a foam developer, e.g., saponin, in a protective colloid, e.g., albumin solution, is added either dry or in solution to a water-gas mixture; alternatively, the foam developer as a powder is mixed with a powder insoluble in water, e.g., china clay.

W. G. CAREY.

Gas analysis recorder. O. RODHE, Assr. to SVENSKA AKTIEBOLAGET MONO (U.S.P. 1,661,049, 28.2.28. Appl., 29.11.22. Swed., 6.12.21. Renewed 14.1.28).—The indicating member of the recorder, operating discontinuously, is controlled by a valve in connexion with a float. Means are provided for opening the valve in one extreme position of the float to release the indicating member, and for closing the valve in the other extreme position of the float to lock the indicating member.

H. HOLMES.

Pyrometer. A. A. GRUBB, Assr. to OHIO BRASS Co. (U.S.P. 1,660,504, 28.2.28. Appl., 6.8.25).—A base-metal couple is enclosed in a tapering metal sheath which is compressed at its lower end to form an intimate contact with the hot junction and inside which the elements are insulated from each other and from the sheath.

A. R. POWELL.

[Continuous] viscosimeters. W. A. NIVLING (B.P. 274,479, 13.7.27. U.S., 13.7.26).—The liquid, of which the viscosity is to be continuously recorded, is pumped at a constant rate into a closed vessel in which air or gas is trapped, and which is provided with a constricted outlet for the liquid, the pressure of the gas is then a measure of the viscosity. A thermometer and excess-pressure safety device may be provided.

B. M. VENABLES.

Hydrometer. C. E. LINEBARGER (B.P. 284,916, 27.5.27).—A hydrometer tube, preferably containing "Chaslyn balls," is adjustably attached to the vent plug of an accumulator (or other vessel) so that it can be used to indicate the level of the liquid as well as its density.

B. M. VENABLES.

Apparatus for testing permeability of materials. A. ABRAMS (U.S.P. 1,660,024, 21.2.28. Appl., 4.5.27).—Material to be tested is releasably clamped in an opening in an exposed lateral face of a container for liquid. The container is secured to a base and has connexions for the supply and discharge of liquid and for maintaining a head of liquid. The clamp is operable without disturbing either connexions or container.

H. HOLMES.

Centrifugal machine. V. REDLICH (U.S.P. 1,663,731, 27.3.28. Appl., 4.5.26. Austr., 19.5.25).—See B.P. 252,339; B., 1926, 775.

Apparatus for treatment of gases. E. STRÖDER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,663,734, 27.3.28. Appl., 29.2.24. Ger., 6.4.23).—See B.P. 213,521; B., 1924, 456.

[Charging hopper for] kilns. E. SOBEK (B.P. 286,956, 15.7.27).

Extraction of vapour, particles, etc. from [engine] gases. D. McC. SHANNON (B.P. 285,928, 26.8.26).

II.—FUEL; GAS; TAR; MINERAL OILS.

Laboratory methods for the determination of the yields of coke and by-products from coal. K. BRÜGEMANN (Brennstoff-Chem., 1928, 9, 59—61; cf. Müschenborn, B., 1927, 576).—The difficulties in the determination of the yields of coke and by-products from the carbonisation of coal in the laboratory so that the results shall be directly applicable to large-scale practice are discussed. The yield of coke is readily determined with accuracy, but the determination of the

yields of tar and benzol from the carbonisation of 15–20 g. of coal is subject to comparatively large errors. Even when the heating conditions are adjusted to approximate closely to those in the coke oven itself the yield of tar may, on account of unavoidable losses, be 20–25% below that obtained on the large scale. The ammonia can be determined sufficiently accurately when account is taken of the ammonium chloride which has condensed with the tar. One calcium chloride tube only is insufficient for drying the gas before absorbing the benzol, and the interposition of a phosphorus pentoxide tube is recommended. For the benzol absorption either two tubes of active charcoal or "potash" bulbs charged with paraffin oil and maintained at 0° may be used. The latter method gives yields more closely agreeing with large-scale practice than the former, which may give values 20–25% too high. The gas yield, like that of the tar, depends on suitable control of the heating, and it is recommended that gas heating be replaced by electrical heating in all laboratory determinations.

A. B. MANNING.

Progress in research on the properties of coke for blast-furnace use. R. A. MOTT (Gas World, 1928, 88, Coking Sect., 44–49).—A review of the progress in oven design and operation followed by preliminary results of research carried out at Sheffield University on behalf of the Blast-Furnace Coke Research Committee, Yorkshire and Derbyshire Coalfields. Cokes of known blast-furnace value have been selected and a number of tests applied designed to measure their mechanical strength and manner of burning under blast-furnace conditions. A shatter test, indicative of the resistance of a coke to break-up on dropping or impact, is carried out by dropping 50 lb. of coke of a size greater than 2 in. four times from a height of 6 ft. on to a steel plate. The residue is sieved and the percentage remaining on a 2 in. screen is called the "shatter index." The mean of three tests is a reliable index of the general blast-furnace value of a coke. Losses obtained in an abrasion test were insufficient to account for the large differences in coke consumption occurring in practice. Combustibility tests comprising the analyses of gas made by the combustion of coke in a furnace at specified rates of air supply show that the better blast-furnace cokes have lower combustibility values than poor blast-furnace cokes, and that easy combustibility is not desirable. The combustibility value gives the percentage gaseous carbon as a percentage of the maximum possible.

H. S. GARLICK.

Melting of fuel ash. K. BUNTE and K. BAUM (Gas-u. Wasserfach, 1928, 71, 97–101, 125–130).—Three points can be distinguished in the melting of an ash sample moulded into a cone: the softening point as shown by the base, the melting point when the cone has settled into a ball, and the fluid point when all shape is lost. The m.p. is lowest with a semi-reducing atmosphere as on a furnace grate, and such should be used. The authors use a cylindrical test specimen 30 mm. long and 30 mm. diam. placed within a vertical heating tube in an electric furnace and having graphite electrodes in contact with the upper and lower ends of the specimen. The upper one is movable, and its position is recorded on a revolving drum so that a record of the length of the specimen

during heating is obtained. Temperatures are recorded by a thermocouple. The specimen is prepared from ash which passes 400-mesh and is moulded in a bronze mould with 10% of dextrin. Finer grinding makes no difference. Uniform heating is essential, but no difference was noticed between a rate of 5° and 8° per min. The specimen showed a slight expansion before softening, then a sharp drop which in some cases was in two stages. A high-silica ash of this type showed a softening interval of no less than 470° due presumably to chemical changes. Possible chemical compounds that may occur in ash are discussed, and the difficulty of forecasting the m.p. from the elementary composition is indicated. The authors' results are compared with those of other workers, and the conclusion is drawn that the most that can be done by such tests is to arrange a series of coals in order of fusibility of the ash.

C. IRWIN.

Melting points of coal ash. D. J. W. KREULEN (Chem. Weekblad, 1928, 25, 192–194).—The finely powdered ash is ignited at a red heat, cooled, moistened with water, moulded into a slender pyramid which is mounted on a clay base, and heated in a Meker muffle, temperatures being recorded by a thermocouple. Details of the manipulation and apparatus are given.

S. I. LEVY.

Conversion of methane into hydrogen and carbon monoxide. F. FISCHER and H. TROPSCH (Brennstoff-Chem., 1928, 9, 39–46).—The effect of 20 different catalysts on the reaction $\text{CH}_4 + \text{CO}_2 \rightarrow 2\text{CO} + 2\text{H}_2$ at 850° has been studied. Copper, iron, molybdenum, and tungsten had a negligible catalytic effect; cobalt and nickel were equally active, giving a 50% conversion under the experimental conditions, but their activity was greatly increased by the addition of alumina, a nickel-alumina catalyst giving a 95% conversion. Porous pot proved to be the most suitable support for the catalyst, other materials breaking down after a time. By mixing a purified coke-oven gas with carbon dioxide in the proportion 4:1 and passing the mixture over one of the more active catalysts at 850–870°, the methane was almost completely converted into hydrogen and carbon monoxide. At the same time the unsaturated hydrocarbons were completely decomposed and the organic sulphur compounds were converted into hydrogen sulphide. By the passage of a mixture of coke-oven gas and water vapour over the same catalysts at 860–930° the reaction $\text{CH}_4 + \text{H}_2\text{O} \rightarrow \text{CO} + 3\text{H}_2$ was made to proceed almost to completion, and a gas was thereby obtained of composition suitable for the benzene synthesis. It was not possible to bring about a complete decomposition of the methane by passage of this mixture over heated coke, even at 1000° with a large excess of water vapour and a slow rate of passage of the gas.

A. B. MANNING.

Continuous vertical-retort practice at Oldbury. J. POULSON (Gas J., 1928, 182, 50–59).—Comprehensive results are given of a series of tests with an installation of Glover-West vertical retorts on certain commercially available coals. The tests comprised the carbonisation of (a) North Staffordshire coals, (b) Derbyshire coals for the production of gas of 450 B.Th.U. gross per cub. ft., (c) Yorkshire coals for the production of gas of 450 and

475 B.Th.U. gross per cub. ft., (*d*) a mixture of North Staffordshire, Derbyshire, and Yorkshire coals with North Wales slack, for the production of gas of 460 B.Th.U. per cub. ft.; a continuous test was made of the operation of a Spencer-Bonecourt horizontal waste-heat boiler, during the period of the above tests. All procedure and analyses follow as closely as possible the routine and methods of the Fuel Research Board, and the results demonstrate the superiority of the continuous vertical system of carbonisation over the intermittent system.

H. S. GARLICK.

Walther Feld [ammonium] polythionate process [for coal gas]. E. TERRES and F. OVERDICK (*Gas- u. Wasserfach*, 1928, 71, 49—53, 81—86, 106—110, 130—136; cf. B., 1912, 430).—The Feld process for the removal of ammonia and sulphur from coal gas by washing with ammonium tetrathionate, work on which ceased with the inventor's death in 1914, has been re-examined, with especial reference to temperature and concentration conditions and reaction velocities. The analytical work, involving the determination of sulphate, sulphite, thiosulphate, and polythionates in aqueous solution, is complex. Thiosulphate may be detected quantitatively by mixing the solution with an equal volume of 10% ammonium molybdate and pouring over concentrated sulphuric acid. A deep blue ring at the surface of contact distinguishes it from sulphite and polythionate. Trithionate may be distinguished from tetra- and pentathionate with mercurous nitrate solution. Mercuric chloride solution also reacts differently with the different acids, and on this methods of determination are based, the free acid liberated being titrated. The regeneration step, in which ammonium thiosulphate in saturated solution is converted by the action of sulphur dioxide into polythionate, was performed at 0°, 25°, and 35°. With small concentrations of sulphur dioxide sulphur separates and sodium sulphite is formed in solution; with higher concentrations the latter disappears. Sodium tetrathionate then separates, whilst the solution contains thiosulphate in decreasing proportions with increase of sulphur dioxide content. The reaction was greatly accelerated by the presence of a trace of potassium arsenite. It affords a convenient means of preparing pure tetrathionate. The partial pressures of these solutions with respect to ammonia, sulphur dioxide, and water vapour at various sulphur dioxide concentrations were determined. That of ammonia was always very small. At 35° the sulphur dioxide partial pressure was less than at 25° for similar concentrations, and was such that burner-gas containing 10—12% SO₂ might be used for the reaction. If ammonia is present in excess in the solution the conversion of thiosulphate into tetrathionate does not occur, the solid salt deposited being either the thiosulphate or the sulphite. If the liquor is now used for washing ammonium sulphide from coal gas, sulphur is precipitated and thiosulphate remains in solution and can be regenerated as usual. A series of solubility curves of the systems ammonium thiosulphate-ammonium sulphite-water and the same with addition of sulphur dioxide is given. The absorption reaction in which ammonia, ammonium sulphide, and ammonium carbonate interact with ammonium tetrathionate with production of thiosulphate is next studied in a similar

way, the temperature being maintained at 25°. A slight initial decomposition of tetrathionate into sulphate and sulphur occurs, followed on the introduction of further ammonia or ammonium salts by conversion into thiosulphate. With ammonia alone the Feld reaction would, therefore, gradually pass into the Burkheiser reaction, proceeding afterwards as indicated above. Under practical conditions there is at least the ammonia equivalent of hydrogen sulphide present. In this case the reaction proceeds smoothly; with excess of hydrogen sulphide more slowly. Partial pressures are much lower than with ammonia alone. The reaction with ammonium carbonate is slow and complicated, the products in addition to ammonium thiosulphate including the sulphate and trithionate. This reaction is, however, considered to be unimportant in practice. Accumulation of ammonium sulphate in solution reduces the solubility of ammonium tetrathionate. It is concluded that the regeneration step is practicable with the help of potassium arsenite as catalyst, and that the absorption proceeds smoothly if ammonia and hydrogen sulphide are equivalent; if the latter is in excess a following treatment with ammonium sulphate will remove it.

C. IRWIN.

Desulphurising action of silica gel and failure of the lamp combustion [method for the determination of sulphur] in the presence of mercaptans. H. I. WATERMAN and M. J. VAN TUSSENBROEK (*Brennstoff-Chem.*, 1928, 9, 37—39; cf. B., 1927, 179).—By shaking kerosene containing sufficient ethyl sulphide to bring its sulphur content to about 1% with 50% of its weight of silica gel about 33% of the sulphur was removed. Similar results were obtained with kerosene solutions of propyl sulphide and the corresponding disulphides, but the adsorptive power of the gel appears to decrease as the mol. wt. of the dissolved sulphur compound increases. The fineness of subdivision of the gel had little effect on the amount of sulphur removed. Treatment with silica gel removed sulphur also from kerosene solutions of mercaptans, but no accurate quantitative results were obtained owing to the failure of the lamp method for determining sulphur in the oils. In the presence of mercaptans this method gave low results, due probably to the formation of asphalt-like substances containing sulphur which were deposited on the wick.

A. B. MANNING.

Reducing power of adsorptive carbons. J. VAŠÁTKO (*Z. Zuckerind. Czechoslov.*, 1928, 52, 221—226).—Decolorising carbons not only have a marked adsorbent capacity for iron salts, but in varying degree they bring about reduction. On shaking them with dilute ferric chloride solution, the adsorptive equilibrium is usually attained in a few minutes; but reduction, although rapid at first, continues slowly for some hours. In experiments described, twelve different carbons, in amounts from 1 to 6 g., were shaken for 3 hrs. at 20° with 100 c.c. portions of 0.5% ferric chloride solution slightly acidified with hydrochloric acid. With 1 g. of the samples the proportion of the total iron reduced ranged from 39% (*Carbo animalis*) to 11% (*Carboraffin H*), except for bone char ("*Spodium K*"), which reduced only 3%. The carbons tested were the same as those employed in previous experiments on the decomposition of sucrose (B., 1928, 29), and it was observed that those

most active in decomposing sucrose were least active in reducing ferric salts, and *vice versa*. J. H. LANE.

✱ **Corrosion in gas-supply practice.** F. C. SMITH (Gas J., 1928, 182, 118—123).—Investigation into the cause of corrosion of gas-supply mains and services showed that iron tubing does not corrode in the absence of liquid water. Oxygen and water will bring about corrosion; the presence of carbon dioxide is not essential for the production of iron oxide, but its presence greatly accelerates the rate of corrosion. In conditions obtaining in a gas main, iron would, in the presence of carbon dioxide, pass into solution in water as ferrous carbonate, and be precipitated by any oxygen in the gas. The finely-divided oxide thus formed is easily transported by gas moving at the usual velocities, and is carried forward to build up an obstruction at any point of resistance. The best methods for preventing corrosion are either to dry the gas, thus rendering the oxygen and carbon dioxide harmless, or to remove the oxygen, thus leaving ferrous bicarbonate in solution. A method of measuring the rate of corrosion of specimens of iron is illustrated, in which the fall in resistance in ohms of a conductivity cell containing iron immersed in water saturated with carbon dioxide is plotted against the time in hours. H. S. GARLICK.

Chemical processes occurring in the hydrogenation of sphagnum under pressure. H. I. WATERMAN and J. N. J. PERQUIN (Brennstoff-Chem., 1928, 9, 57—59; cf. B., 1926, 974).—A mixture of 50 g. of sphagnum and 150 g. of oil (Edeleanu extract) was heated at 450° with hydrogen under pressure. Comparison of the product with that from a blank experiment on the Edeleanu extract alone showed that the yield of oil from the hydrogenation of the sphagnum amounted to over 14%, calculated on the dry, ash-free material; in addition, considerable quantities of carbon monoxide, carbon dioxide, and water were evolved. After being kept for some days the oil deposited an asphalt-like substance which was insoluble in xylene. A similar phenomenon has been observed in oils produced by the berginisation of coal. Precautions should be taken to prevent such a deposition during hydrogenation, which would lead to the formation of coke in the bomb. It is advisable, therefore, to work in a stable fluid medium, to avoid too high a temperature and too long a period of heating, and if possible to remove the light-boiling products as they are formed. Vegetable oils are hydrogenated under pressure without difficulty, owing to their low oxygen content. A. B. MANNING.

✱ **Ultramicroscopical examination of asphalt.** F. J. NELLENSTEYN and J. P. KUIPERS (Chem. Weekblad, 1928, 25, 190—192).—The examinations were carried out at a magnification of 1000, with cedar oil immersion. Artificial asphalts do not readily show active motion, but after shaking with Portland cement give the same results as the natural asphalts; the colloidal structure of asphalts is not due to the inorganic constituents. The activity depends on the solubilities and on the surface tensions of both the asphalt and the solvent; the best results are obtained when the surface tension of the solvent is somewhat higher than that of the solute. Hence the results with a solution of coal tar in carbon

disulphide are not comparable with those obtained with a solution of asphalt in the same solvent; for strict comparison, a solvent of surface tension about 30 should be used with asphalt, and of about 42 with coal tar.

S. I. LEVY.

Composition of the products obtained in the synthesis of petroleum. F. FISCHER and H. TROPSCH [with H. KOCH] (Brennstoff-Chem., 1928, 9, 21—24).—The gasol, benzene, and petroleum fractions of the synthetic petroleum obtained by the catalytic reduction of carbon monoxide at ordinary pressures (B., 1926, 475) have been examined. The gasol consists of ethane, propane, and butane, together with the corresponding olefines; after 24 hrs. operation an iron-copper catalyst gave a product containing 26.5% of olefines, whilst after 65 hrs. the proportion rose to 36%. The benzene (initial b.p. 59°), of which 95% distilled below 185°, had d_4^{20} 0.7135, iodine value 161, and contained 65% of unsaturated hydrocarbons. The latter are probably simple olefines, the benzene being stable to light and air and showing no tendency to discolour or deposit gums. By varying the experimental conditions it is possible to obtain more highly saturated benzenes if desired. *n*-Octane, *n*-nonane, and isononane were identified in the saturated hydrocarbons remaining after exhaustive extraction with fuming sulphuric acid of a fraction of boiling range 125—150°. The iodine value of the petroleum fraction varied considerably with the experimental conditions, *e.g.*, from 8.7 to 64.7. It increased with the gas velocity and with the age of the catalyst. The water-soluble organic products of the reaction amounted at most to 2% of the total products, and consisted principally of aldehydes and ketones. In addition to theories of the mechanism of the reaction previously suggested (*loc. cit.*), the possibility of the formation of hydrocarbons of high mol. wt. which subsequently undergo a type of cracking process is briefly discussed.

A. B. MANNING.

Alumina gel as a desulphurising agent in petroleum refining. J. K. CHOWDHURY and R. C. BAGCHI (J. Indian Chem. Soc., 1928, 5, 111—125).—Alumina gel (water content 6%), activated under optimum conditions by the ignition of aluminium hydroxide at 350—400°, absorbs a large number of sulphur compounds from their solutions in petroleum, and more especially those in which the sulphur is contained in an oxidised form. Thus a preliminary oxidation of solutions of bivalent sulphur compounds by the passage of air in the presence of alumina gel leads to more complete adsorption (cf. G.P. 405,850; B., 1925, 179). Adsorption is also increased by the use of large amounts of very finely-divided alumina gel, but these modifications involve loss of petroleum and difficulties of filtration. Admixture of activated carbon with the gel yields an adsorbent which is more efficient than either of its components. More pronounced results are obtained by the use of a mixture of alumina and silica gels, prepared by the precipitation of alumina gel on silica gel followed by ignition at 230—235°. No increase in activity is obtained by the precipitation of the alumina on an inert surface such as powdered glass or pumice. Alumina gel may be reactivated by treatment with superheated steam and air. The application

of Freundlich's equation in the examples in which the pure gel is used show that the action is one of true adsorption.

G. A. C. GOUGH.

Determination of aromatic and unsaturated hydrocarbons in light petroleum. E. H. RIESENFELD and G. BANDTE (Brennstoff-Chem., 1928, 9, 30).—Kattwinkel (B., 1928, 78) has overlooked a later publication of the authors in which some of their previous results are corrected (cf. Auto-Technik, 1927, 16, 7).

A. B. MANNING.

Standardisation of oil testing. H. SCHLÜTER (Chem.-Ztg., 1928, 52, 261—264).—An apparatus is described by the use of which uniform results are obtained in the open flash-point test, the chief cause of variations in the existing method being the lack of precision in placing the thermometer with relation to the bottom of the vessel.

S. I. LEVY.

Ammonium sulphate. SCHRAMM, also WEINDEL.—See VII. **Concrete in gas works.** ORTHAUS.—See IX. **Active carbons.** MRASEK, also NOSEK.—See XVII.

PATENTS.

Coke ovens. N. V. SILICA EN OVENBOUW MIJ., Assees. of C. OTTO & Co. G.M.B.H. (B.P. 272,942, 16.6.27. Ger., 16.6.26).—In coke ovens with adjacent upflow and downflow heating flues connected at the top and bottom, the openings at the bottom are provided with regulating devices, *e.g.*, sliding bricks, which can be adjusted from the outside, so that the supply of waste gas can be readily controlled.

A. B. MANNING.

Coking retort oven. J. VAN ACKEREN, Assr. to KOPPERS Co. (U.S.P. 1,660,658, 28.2.28. Appl., 13.10.22).—A coking retort oven is made up of a number of coking chambers having heated walls constituted alternately of intercommunicating vertical combustion flues and outflow chambers, which communicate with a system of recuperators and regenerators.

C. O. HARVEY.

Carbonising and gas-making apparatus. P. PLANTINGA (CLEVELAND TRUST Co., Exors.) (U.S.P. 1,663,522, 20.3.28. Appl., 21.12.22).—The apparatus consists of a carbonising chamber surrounded by a jacket, both of which communicate with a gas producer.

C. O. HARVEY.

Apparatus for treatment of marine algæ. G. J. B. CHAMAGNE (B.P. 275,998, 11.8.27. Fr., 11.8.26).—Marine algæ are carbonised in a furnace in which they slide under their own weight down an inclined bed as they burn, the supply of air being so regulated that only incomplete combustion at a low temperature occurs, and loss of volatile iodine and bromine compounds is avoided. The material passes from the inclined bed to a horizontal bed where the carbonisation is completed, and thence to an extinguisher arranged below the inclined bed of the furnace, in which the carbonised material cools. Water tubes within the carbonisation chamber provide means for cooling the latter and at the same time recovering part of the heat liberated therein.

A. B. MANNING.

Production of high-grade products from raw coal. F. L. KÜHLWEIN, Assr. to CHEM. FABR. IN BILLWÄRDER VORM. HELL & STHAMER A.-G. (U.S.P.

1,660,009, 21.2.28. Appl., 21.3.27. Ger., 30.12.26).—A mixture containing coal and clayey ingredients is separated by dispersing the clayey matter with a weakly alkaline solution of a xanthate.

C. O. HARVEY.

Preparation of liquid fuels for combustion. F. B. DEHN. From GASONE CORP. (B.P. 286,386, 9.12.26).—An emulsion of the fuel with an expansible medium is produced by passage through a system of pumping cylinders and mixing chambers, wherein the initial pressure of mixing is about 100 lb./in.², and the final pressure may be as high as 3000 lb./in.² *E.g.*, an emulsion of oil, water, and air of predetermined composition may be produced, the pressure being released only at the moment of injection into the cylinder of an internal-combustion engine.

C. O. HARVEY.

Drying of fuel gases. C. COOPER and W. C. HOLMES & Co. (B.P. 285,936, 21.10.26).—The gases are passed through a washer containing a hygroscopic liquid, and means are provided for circulating the liquid independently through (a) a heat-exchanger in order to dissipate the heat developed during absorption of moisture from the gas, and (b) an evaporator for reconcentrating the liquid.

A. B. MANNING.

Purification of illuminating gas. SOC. DU GAZ DE PARIS (B.P. 278,700, 29.9.27. Fr., 11.10.26).—The gas is passed first through a mixture of a solution of a ferrous salt with milk of lime, and then through a suspension of the powdered solid, consisting of ferric hydroxide mixed with sulphur and calcium salts, obtained by filtering the product from the first stage and oxidising the residue in air. The hydrocyanic acid, together with part of the hydrogen sulphide, is removed in the first stage, and the remaining hydrogen sulphide in the second. The suspension used in the second stage is regenerated at intervals by the passage of air.

A. B. MANNING.

Simultaneous absorption of ammonia and hydrogen sulphide from industrial gases. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 285,999, 26.11.26).—Ammonia and hydrogen sulphide are recovered from industrial gases by simultaneous absorption in an aqueous ammonium sulphite-bisulphite solution with an $\text{NH}_3 : \text{SO}_2$ ratio between 1.2:1 and 1.75:1. The ammonia is converted into sulphite and the hydrogen sulphide principally into thiosulphate. The process can be carried out in two stages, using a solution of lower $\text{NH}_3 : \text{SO}_2$ ratio in the first stage, and one of the highest permissible ratio in the second. It is an advantage to maintain the temperature of the solution at 60–70° in the first stage, and to cool the gas before treatment in the second stage.

A. B. MANNING.

Treatment of gases. O. L. BARNEBEY, Assr. to AMER. SOLVENT RECOVERY CORP. (U.S.P. 1,660,642, 28.2.28. Appl., 2.1.20. Renewed 11.5.27).—The gas is adsorbed by means of activated carbonaceous material, whence it is expelled in a closed system and liquefied.

C. O. HARVEY.

Manufacture of a decolorising carbon. C. H. CHANDLER (U.S.P. 1,663,000, 20.3.28. Appl., 18.6.20).—Sugar-cane pith is softened and carbonised, and the product extracted.

L. A. COLES.

Distillation of solid bituminous materials. E. PIRON (B.P. 275,178, 13.6.27. U.S., 27.7.26).—Coal is

distilled by heating a layer $\frac{1}{4}$ to $\frac{3}{4}$ in. in depth on a surface the temperature of which is maintained at 590–790°, the volatile decomposition products being evolved at 427–538°, and carbonisation being complete in about 5 min. A high yield of tar is obtained, having a larger proportion of valuable light constituents and low-boiling tar acids than tars produced by processes using thicker layers of coal. A. B. MANNING.

Manufacture of agents for emulsifying, cleansing, wetting, etc. I. G. FARBERIND. A.-G. (B.P. 271,474, 17.5.27. Ger., 19.5.26).—A mixture of a brown coal tar oil and an aromatic or hydroaromatic hydrocarbon is sulphonated, preferably by the addition of chlorosulphonic acid to the cooled mixture, and the sulphonic acid isolated as the sodium salt. A. B. MANNING.

Cracking of hydrocarbon [oils]. J. E. BELL and E. W. ISOM, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,660,856, 28.2.28. Appl., 14.3.25).—The oil is circulated and heated to a cracking temperature under pressure in two stages, the evolved vapours are refluxed, and the condensate is returned to the first stage. The heat is supplied by indirect contact with hot gases, a portion of which is recirculated after mixing with the heating gases in the first stage. C. O. HARVEY.

Cracking of hydrocarbons. E. W. ISOM, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,661,073, 28.2.28. Appl., 15.5.25).—A stream of oil is passed once only through a long heating conduit heated by contact with the products of combustion from a furnace which pass over the outer surface of the conduit in a direction generally opposite to that of the flow of oil within the conduit. The temperature of the gases leaving the heater is below that of the heated oil flowing out from the conduit. A part of the residual heating gases is recirculated in admixture with the fresh hot products of combustion. C. O. HARVEY.

Combined distillation and cracking process [for hydrocarbons]. M. J. TRUMBLE (U.S.P. 1,659,930, 21.2.28. Appl., 8.9.24).—Raw material, capable of giving off vapours too heavy for use alone in a "prime mover," is supplied to a heated retort, and the evolved vapours are mixed with those obtained from an oil-cracking still before use. C. O. HARVEY.

Cracking of oil. R. S. PERSHING (U.S.P. 1,661,196, 6.3.28. Appl., 28.10.24. Renewed 21.7.27).—The oil is cracked by spraying it upon an electrically-heated metallic drum situated within a still. C. O. HARVEY.

Conversion of high-boiling hydrocarbons into low-boiling hydrocarbons. Motor fuel and refining of oils. L. EDELEANU, Assr. to ALLGEM. GES. F. CHEM. IND. (U.S.P. 1,661,565—6, 6.3.28. Appl., [A] 28.6.26, [B] 24.3.27).—(A) Before subjecting the oil to the usual cracking process the sulphur, nitrogen, and asphaltic compounds are removed by extracting the crude oil with liquid sulphur dioxide. (B) Commercial petrol is redistilled at 160°, and the non-volatile portion treated with liquid sulphur dioxide to remove the aromatic and unsaturated hydrocarbons, which are recovered by distilling off the sulphur dioxide, and added to the portion of the original oil distilling below 160° to obtain a fuel which has a much reduced tendency to knock.

The residue from the sulphur dioxide treatment is purified to obtain a high-grade kerosene.

A. R. POWELL.

Separation of oils. J. K. PIERCE, Assr. to W. S. PIERCE (U.S.P. 1,660,235, 21.2.28. Appl., 5.4.26).—An apparatus for the continuous chemical treatment of oils and for separating the oil from the reagent consists of two interconnected vertical tanks having a constant-level device whereby the upper surface of the liquid reagent is maintained on a level with the pipe connecting the two tanks, the oil spaces being above this level. The inlet for the oil is situated at the base of the first tank, and immediately above this inlet is a baffle plate which is submerged in the chemical solution. The interconnecting tube is fitted with a trap for receiving the chemical solution, and is also adapted to carry bottom settlings from the first to the second tank. The weakened chemical solution is circulated back, through a heater, from the second tank to the first, where it meets the incoming oil in counter-current. The treated oil may be withdrawn from the top of both tanks.

C. O. HARVEY.

Treatment of emulsified oils. C. C. MONGER, Assr. to J. S. J. LYELL (U.S.P. 1,660,230, 21.2.28. Appl., 27.11.25).—Oil is separated from a foreign liquid by projecting the mixture at a high velocity against a stationary solid object which is entirely immersed in water. C. O. HARVEY.

Mixing of [acid] sludge. R. BEATTIE, Assr. to TEXAS PACIFIC COAL & OIL Co. (U.S.P. 1,660,248, 21.2.28. Appl., 6.6.27).—Acid sludge and similar residues are mixed with liquid hydrocarbons, and the mixture flows through a passageway wherein it is agitated with sufficient violence to produce a homogeneous liquid mixture. C. O. HARVEY.

Transference of gases and [hydrocarbon] vapours. G. E. FOX, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,659,948, 21.2.28. Appl., 9.12.25).—Hydrocarbon vapours are transferred from a heating coil to an enlarged reaction chamber (at a pressure of about 350 lb./in.²) by passing the product from the coil partly in vapour phase into a conduit at a pressure not exceeding 50 lb./in.², introducing into the conduit in the direction of travel a stream of gas at a pressure of about 1000 lb./in.², constricting the flow for a brief period, and then allowing expansion into the enlarged chamber. C. O. HARVEY.

Preparation of a [hydrocarbon] oil composition. Treatment of hydrocarbon residues. R. T. GOODWIN, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,660,294—5, 21.2.28. Appl., [A] 20.1.25, [B] 25.4.25. Renewed [A] 12.10.27).—Heavy oils or cracked hydrocarbon oil residues, liable to form carbonaceous sediments on being kept, are mixed with (A) an oil containing a small percentage of mineral acid or (B) small amounts of an acidic substance in non-sludge-forming concentration, whereby relatively rapid precipitation of the sediment is effected, and the clear liquid is withdrawn. C. O. HARVEY.

Purification of petroleum oils for removal of sulphonated compounds. R. C. MORAN, Assr. to VACUUM OIL Co. (U.S.P. 1,659,782, 21.2.28. Appl.,

23.2.27).—The acid-treated petroleum oil is extracted with ethylene glycol. C. O. HARVEY.

Apparatus for discharging gas or coke retorts. C. HOLLANDER and GIBBONS BROS., LTD. (B.P. 286,846, 5.2.27).

Washing of minerals (B.P. 285,987). **Liquid-fuel filter** (B.P. 279,382). **Treatment of fluids** (B.P. 284,758). **Gas analysis recorder** (U.S.P. 1,661,049).—See I. **Lead tetra-alkyl** (U.S.P. 1,661,809 and 1,661,810). **Lead tetra-ethyl** (U.S.P. 1,664,021).—See VII. **Briquettes** (B.P. 286,334).—See IX. **Rubber goods** (B.P. 286,171).—See XIV.

III.—ORGANIC INTERMEDIATES.

Catalytic reduction of nitrobenzene to aniline in the gaseous phase. G. B. SEMERIA and M. MILONE (Annali Chim. Appl., 1928, 18, 68—77).—The reduction of nitrobenzene vapour to aniline by means of hydrogen in presence of coppered or silvered asbestos is exothermic, and the best yield is obtained at temperatures not exceeding 350° with copper and at 400° with silver. The yield at first increases rapidly and then remains constant as the quantity of catalyst increases. The influence of increase of the amount of hydrogen used on the yield becomes larger as the temperature is raised and as the catalyst is increased in quantity. The yield is not changed much by variation in the hourly supply of nitrobenzene. The catalyst retains its initial activity for months. The aniline obtained in presence of copper is always slightly coloured by the presence of traces of nitrobenzene, but the silver catalyst gives pure aniline in quantitative yield. T. H. POPE.

PATENTS.

Production of acetone. K. RÖKA, Assr. to HOLZVER, KOHLUNGS-IND. A.-G. (U.S.P. 1,663,350, 20.3.28. Appl. 12.12.26. Ger., 10.12.24).—Ethyl alcohol and water vapour are made to react in the presence of a catalyst at 250—650°. H. ROYAL-DAWSON.

Manufacture of unsaturated hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 264,827, 17.1.27. Ger., 19.1.26).—Unsaturated aliphatic hydrocarbons are produced by passing saturated aliphatic hydrocarbons, and in particular methane, at atmospheric or higher pressures, over a catalyst, e.g., salts or hydroxides of the alkaline-earth metals, magnesium, or beryllium, compounds of selenium, tellurium, or thallium, or active carbon or silica, or mixtures of these, at 700—1000°, at a sufficiently rapid rate to preclude further condensation of the primary products into higher-molecular, e.g., aromatic, hydrocarbons. A. B. MANNING.

Guanidine compound. R. V. HEUSER, Assr. to AMER. CYANAMID Co. (U.S.P. 1,663,128, 20.3.28. Appl., 24.4.26).—Substituted guanidines having at least one aminic hydrogen atom are treated with carbon disulphide and then oxidised to a diguanylthiuram disulphide; in particular *N*-phenyl-*N*-ethylguanidine gives a phenyl-ethylguanylidithiocarbamate which is oxidised to diphenyl-diethylguanylthiuram disulphide. C. HOLLINS.

Manufacture of substituted guanidines. W. CARPMAEL. From CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 285,932, 16.10.26, and Addn. B.P.

286,196, 15.10.26).—(A) *S*-Alkylisothiocarbamides react with liquid or solid, basic, primary or secondary monoamines in approximately equimolecular proportions to give guanidines; excess of amine or addition of alkali is unnecessary. The following guanidines are described: *N*-β-hydroxyethyl-, *N*-β-acetoxyethyl-, *N*-methyl-*N*-β-hydroxyethyl- (hydrochloride, m.p. 78°; hydrobromide, m.p. 103°; picrate, m.p. 166°; picrolonate, decomp. 236—237°), *N*-methyl-*N*-β-acetoxyethyl-, *N*-ethyl-*N*-β-hydroxyethyl- (picrate, m.p. 158°; hydrochloride), *N*-β-hydroxyethyl-*N*-isoamyl- (picrate, m.p. 117—118°; hydrochloride), *NN'*-dimethyl-*N'*-β-hydroxyethyl- (picrate, picrolonate, hydrochloride, hydrobromide, hydriodide), *N*-β-diethoxyethyl- (hydrobromide, m.p. 53—54°; hydriodide, m.p. 60°; picrate, m.p. 201°), *N*-methyl-*N*-ββ-dihydroxyethyl- (picrate, m.p. 116°), *N*-methyl-*N'*-ββ-dihydroxyethyl- (picrate, m.p. 107°), *N*-2-pyridyl- (hydrobromide), *NN''*-di-*p*-nitrobenzoyl-*N'*-*N'*-diethyl-, m.p. 181°, *NN''*-dicarbethoxy-*N'*-benzyl-, m.p. 35°, *NN''*-dibenzoyl-*N'*-methyl-*N'*-β-hydroxyethyl-, also 1-guanylpiperidine (hydrochloride, m.p. 166°; hydrobromide, m.p. 122°; picrate, m.p. 248°), guanylpyrrolidine (picrate, m.p. 211°; hydrochloride; hydrobromide; hydriodide), and 4-guanidino-1-phenyl-2:3-dimethyl-5-pyrazolone (from antipyrine). (B) In place of *S*-alkylisothiocarbamides cyanamide may be used for the manufacture of the guanidino-alcohols; e.g., cyanamide and β-methylaminoethyl alcohol give *N*-methyl-*N*-β-hydroxyethylguanidine. C. HOLLINS.

Production of mercaptans of the furfuryl series.

E. C. R. MARKS. From INTERNAT. NAHRUNGS- U. GENUSSMITTEL A.-G. (B.P. 286,152, 25.7.27).—*Difurfuryl disulphide*, m.p. 10—10.5°, b.p. 112—115°/0.5 mm., obtained by the action of aqueous-alcoholic ammonium or sodium hydrosulphide on furfuraldehyde, is reduced by aluminium amalgam and water, or sodium and alcohol, to *furfuryl mercaptan*, b.p. 45—47°/12 mm. (silver, lead, and zinc salts; *p*-nitrobenzoate, m.p. 76—77°). 5:5'-*Dimethyldifurfuryl disulphide*, m.p. 42°, b.p. 150°/0.5 mm., 5-methylfurfuryl mercaptan, b.p. 61—62°/13 mm. (*p*-nitrobenzoate, m.p. 82—83°), 5:5'-bishydroxymethyl-difurfuryl disulphide, m.p. 136—137°, 5-hydroxymethylfurfuryl mercaptan, b.p. 115—118°/0.5 mm., 5:5'-bismethoxymethyldifurfuryl disulphide, m.p. 36—37°, and 5-methoxymethylfurfuryl mercaptan, b.p. 78—80°, are similarly prepared. The compounds are used to give coffee aroma. C. HOLLINS.

Manufacture of isatins of the naphthalene series.

W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 286,358, 1.12.26).—Naphthyloxamic chlorides are converted into naphthhisatins by warming with an acid condensing agent (aluminium chloride, ferric chloride, etc.) in presence or absence of a solvent (nitrobenzene). α- and β-Naphthhisatins, 9-chloro-α-naphthhisatin, m.p. 239°, 6:9-dichloro-α-naphthhisatin, m.p. 275—276°, 9-chloro-5-bromo-α-naphthhisatin, m.p. 279°, and 9-methoxy-α-naphthhisatin, m.p. 264°, are thus prepared from the corresponding oxamic chlorides. C. HOLLINS.

Manufacture of diazosulphamic acids of the cyclic series. I. G. FARBENIND. A.-G. (B.P. 266,388, [A] 22.2.27. Ger., 22.2.26, and Addn. B.P. 269,582, [B] 14.4.27. Ger., 15.4.26).—(A) By treating sulphamic acids from aromatic polyamines with nitrous acid, one

sulphonic group may be removed, giving diazosulphamic acids, which may be coupled in the usual manner and subsequently hydrolysed to aminoazo compounds. Examples of polyamines used are *o*- and *p*-phenylenediamines, *m*-tolylenediamine, benzidine, 1:5- and 1:8-naphthylenediamines. (b) Instead of polyamines, nitroarylamines are converted into sulphamic acids, and then reduced in neutral solution and diazotised. Examples are *m*- and *p*-nitroanilines, and 6-nitro-*o*-toluidine.

C. HOLLINS.

Manufacture of vanillin. SOC. ANON. PROD. CHIM. COVERLIN, Assees. of R. H. BOTS (B.P. 271,818, 2.2.27. U.S., 25.5.26).—See U.S.P. 1,643,805; B., 1927, 924.

Manufacture of sulphonamides of 2:3-hydroxynaphthoic arylamides. J. B. PAYMAN and H. WIGNALL, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,663,725, 27.3.28. Appl., 15.9.27. U.K., 20.9.26).—See B.P. 281,795; B., 1928, 118.

Sulphonated fatty acids (B.P. 263,117).—See XII. **Acetone and butyl alcohol** (B.P. 268,749). **Dihydroxyacetone** (B.P. 282,347). **Acetone from vinasses etc.** (B.P. 277,932).—See XVIII. **Polyamino-compounds** (B.P. 286,087). **2-Chloropyridine** (B.P. 281,050).—See XX.

IV.—DYESTUFFS.

PATENTS.

Manufacture of [vat] dyes [of the dibenzanthrone series]. R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 286,323, 24.9.26).—Di- and tetrachlorodibenzanthrones (B.P. 278,834; B., 1928, 9) are condensed with glycols (ethylene glycol, trimethylene glycol, glycerol) in presence of alkali (potassium carbonate) and copper in nitrobenzene to give reddish-blue vat dyes.

C. HOLLINS.

Manufacture of azo dyes and their chromium compounds. I. G. FARBENIND. A.-G. (B.P. 279,429, 7.10.27. Ger., 22.10.26).—1:2-Aminonaphthol or one of its diazotisable derivatives is diazotised and coupled with 1:8-naphtholsulphonic acid. The product from 1:2:4-aminonaphtholsulphonic acid gives a red on wool, becoming grey on chroming, or if first converted into its chromium compound it dyes wool greenish-blue. The product from nitrated diazotised 1:2:4-aminonaphtholsulphonic acid, after boiling with chromium formate, dyes wool in fast black shades. C. HOLLINS.

Manufacture of new solid diazo salts. I. G. FARBENIND. A.-G. (B.P. 265,985, 11.2.27. Ger., 12.2.26).—*p*-Chlorobenzenesulphonic acid (or its salts) when added to a diazotised nitroaniline, chloronitroaniline, chloro- or nitro-toluidine, nitroaminophenyl alkyl ether, *o*-aminophenyl aryl ether, or a non-sulphonated derivative of one of these, precipitates a crystalline, water-soluble, stable diazonium *p*-chlorobenzenesulphonate, which may be dried at a moderate temperature (cf. B.P. 273,352; B., 1927, 352).

C. HOLLINS.

Manufacture of new indigoid dyes. W. CARPMAEL. I. G. FARBENIND. A.-G. (B.P. 286,359, 1.12.26).—9-Halogeno- α -naphthisatins (cf. B.P. 286,358; B., 1928, 327), or their 2-anils or 2-chlorides are condensed with the usual reactive methylene compounds to give fast violet

to black vat dyes. Examples are 9-chloro- α -naphthisatin 2-chloride with 6-chloro-4-methylthioindoxyl (violet), 5:8-dichloro- α -naphthisatin with β -naphthathioindoxyl (olive), and 9-chloro-5-bromo- α -naphthisatin 2-chloride with 1-acetylindoxyl (greyish-blue). C. HOLLINS.

Preparation of biological stains, bactericidal agents, etc. R. W. FRENCH and W. C. HOLMES, Assrs. to W. F. KEOHAN (U.S.P. 1,661,568, 6.3.28. Appl., 9.2.26).—Reactive forms of basic dyes, *e.g.*, their leucobases or acetyl derivatives, are caused to interact either in solution or in a molten mass with cyclic compounds containing an acid or phenolic group. A. R. POWELL.

Red disazo dyes for cotton printing. W. NEELMEIER and H. SCHNEIDER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,663,950, 27.3.28. Appl., 23.3.25. Ger., 4.4.24).—See B.P. 231,885; B., 1925, 954.

Manufacture of tetrakisazo dyes. NEWPORT CO. (B.P. 265,553, 20.12.26. U.S., 6.2.26).—See U.S.P. 1,615,551; B., 1927, 246.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Pulping of pine wood by the sulphite process. II. E. HÄGGLUND (Cellulosechem., 1928, 9, 38—43; cf. B., 1927, 294).—Pine wood, after extraction with benzene or ether, still contains substances soluble in acetone or alcohol which prevent uniform pulping by the ordinary sulphite process. When boiled with concentrated sodium bisulphite solution, pine wood affords a product which is satisfactory in pulping properties and in yield, but, due to its high resin content, is unsuitable for paper manufacture. The "Keebra" method of wood digestion (cf. Clark, B., 1926, 781) gives very poor results when applied to pine wood. Pretreatment with alkali at 105° results in the formation of formic and acetic acids; thus, with saturated lime water, about 1% of acetic acid (on the weight of wood) is obtained. Pine wood treated with alkali yields in the sulphite process a product of normal lignin content, but of high ash content, whilst Scotch pine wood extracted with benzene and acetone yields a poorer product after treatment with lime water than before. Extraction with benzene alone and lime treatment gives a very inferior product, and pretreatment with hydrochloric acid is also disadvantageous. Treatment of unextracted Scotch pine wood with alkali at 105° yields in the sulphite process a badly pulped product which contains much lignin, ash, and resin.

W. J. POWELL.

Sulphite cooking. W. H. BIRCHARD (Canad. Chem. Met., 1928, 12, 31—34).—The more important factors in sulphite cooking are discussed, and it is suggested that the lime present in the cooking liquor acts not only as a neutralising agent for acids formed during digestion, but also later, when sulphonation is complete, as an alkaline hydrolysing agent, the normal calcium sulphite dissociating at about 128° into free lime and sulphur dioxide. That calcium hydroxide is precipitated as such at about 128° (though it recombines to form calcium sulphite on cooling, and is found in this form on the fibre) is shown by an analysis of the precipitate obtained by heating sulphite liquor (free SO₂ 3.84%, combined SO₂ 1.31%) to 137° and under 80 lb./in.², and drawing off as much as possible of the supernatant liquor while

the temperature is still above that at which the lime and sulphur dioxide recombine to form calcium sulphite.

D. J. NORMAN.

Degradation of cellulose by sulphite acid. W. H. BIRCHARD (J.S.C.I., 1928, 47, 49—52 T).—Experimental cooks by the sulphite process on cellulose in various forms, viz., bleached and unbleached wood pulps, straw pulp, bleached and unbleached rag stock, and pure and raw cotton, show that, in general, the higher the α -cellulose content of the untreated pulp the greater is the degrading action of sulphite acid: *e.g.*, unbleached sulphite pulp (85% of α -cellulose) lost only 1% of α -cellulose on digestion, whilst pure cotton (99.2% of α -cellulose) lost 28.1% of α -cellulose. This effect may be due to the protective action of carbohydrates, principally sugars, which are closely associated with wood cellulose, but are not present in cotton. In all cases the pulps showed a considerable loss of fibre length (69—88%), but were unaltered as regards the diameter of the fibres. The depolymerisation of the cellulose molecule into β - and γ -cellulose, caused by sulphite acid under ordinary cooking conditions, appears to reach an equilibrium, further depolymerisation being arrested until either the β - or the γ -cellulose is destroyed. This theory is supported by the observation that β -cellulose is only slightly affected when cooked by the sulphite process, whilst 100% α -cellulose is degraded to such an extent that the determination of α -cellulose in the cooked product is impracticable.

D. J. NORMAN.

Determination of α -, β -, and γ -cellulose. G. PORRVIK (Papier-Fabr., 1928, 26, 81—85, 120—124, 133—139, 151—157, 179—183).—The influence of various factors (*e.g.*, ratio of sodium hydroxide to cellulose used; moisture content and condition of swelling of the material; addition of alcohol to, and concentration of, the sodium hydroxide used) on results obtained by the ordinary methods of analysis has been determined. Results vary with the degree of kneading of the material with sodium hydroxide, time of treatment, temperature of drying, etc.; a personal factor is also involved. The determination of α -, β -, and γ -cellulose by a titration method is described, an essential feature of which is the washing of the material previously treated with 18% sodium hydroxide with a solution of the same concentration instead of with water as in the Jentgen method. Results for the α -cellulose content depend on the concentration of sodium hydroxide used, a minimum value being found for an 11% solution, for which also the β -cellulose value is a maximum, whilst for γ -cellulose the results are approximately constant over the range 11—24%; the values for the α - and β -portions, however, are also dependent on the degree of swelling of the material. In Jentgen's method (and others) a change in concentration from 18 to about 9.5% is involved, hence values near to the minimum for α -cellulose are obtained, the exact result depending on several experimental factors, but being about 2% lower than is obtained by the titration method. The former method also gives lower α values the greater the concentration of the sodium hydroxide solution, whilst the reverse is the case for the latter for solutions more concentrated than 11%. Residues having different properties (*e.g.*, copper number) are obtained by treatment

of samples of the same material with either concentrated or dilute alkali so that in both cases the same amount is dissolved. Through the action of sodium hydroxide a slow change of β - into γ -cellulose takes place, prolongation of the time of reaction giving decreased α , approximately constant β , and enhanced γ values. Two new gravimetric methods for determining α -cellulose and a volumetric method for the determination of gum number are elaborated.

B. P. RIDGE.

Determination of α -cellulose. C. G. SCHWALBE (Papier-Fabr., 1928, 13, 189—198).—Different methods for determining α -cellulose have been examined and compared, considerable variations in results on the same material being obtained. The determination of this substance is a conventional procedure owing to the impossibility of separating wood, gum, resin, fat, lignin residues, etc. from the cellulose. Comparable results are only obtainable, therefore, by following exactly an agreed procedure. A large number of factors, such as sampling, drying, mercerisation, swelling, temperature, dilution of the sodium hydroxide, filtration, washing, etc., affect the values given. Bubeck's method was found to give somewhat more satisfactory results than that due to Jentgen, whilst the use of a thermostat and washing with sodium hydroxide are unnecessary complications of these methods.

B. P. RIDGE.

Determination of the copper number of paper. B. W. SCRIBNER and W. R. BRODE (Papier-Fabr., 1928, 26, 130—132).—Paper (1.5 g.) in the form of meal is heated with Fehling's solution at 100° for 30 min., collected on a filter while hot, washed, transferred to a beaker, and treated with a solution of sodium molybdate and phosphoric acid in sulphuric acid. The pulp is again washed on a filter with cold water until all the molybdenum-blue has disappeared, and the blue liquid is titrated with standard potassium permanganate solution, the blue colour disappearing just before the end-point. The copper number of the material is calculated from the permanganate titration in the usual manner.

B. P. RIDGE.

Synthesis of α -pine lignin. P. KLASON (Papier-Fabr., 1928, 26, 208—211; Ber., 1928, 61, [B], 171).—See A., 1928, 277.

Hydrogenation of sphagnum. WATERMAN and PERQUIN.—See II. **Hygiene and artificial silk manufacture.** POMILIO.—See XXIII.

PATENTS.

Manufacture of hard, grainless fibre products. W. H. MASON, Assr. to MASON FIBRE Co. (U.S.P. 1,663,505, 20.3.28. Appl., 18.9.25).—Wood or woody material is disintegrated almost wholly into the fibrous state and converted into a dense, grainless product. During manufacture little loss of material occurs and moisture is almost completely eliminated.

F. G. CLARKE.

Manufacture of artificial silk and like filaments. K. HAGIWARA (B.P. 286,086, 1.3.27).—The speed of filtering or spinning of colloidal solutions of artificial silk is considerably accelerated by utilising an electric current of high voltage, *e.g.*, a high-frequency current from an induction coil, to produce a cataphoresis effect. Thus, in spinning, the passage of such current (frequency

4800 cycles/min.) regulated to a spark gap of 5–10 mm. between an anode placed in the spinning nozzle and a cathode in the coagulating bath renders it possible to increase the spinning speed by 100% or more, and to obtain filaments of 25, 12–15, and 8–10 deniers at speeds of 15–20, 30, and 45 m./min., respectively, from a nozzle ordinarily used for filaments of 6–7 deniers. Further, the gases which collect on the surface of the filament during coagulation are decomposed by the electrical discharge and rendered innocuous. The silk is more uniform in thickness and shows improved physical properties due to the parallel arrangement of the colloid particles.

D. J. NORMAN.

Manufacture of viscose artificial silk. H. J. J. JANSSEN, and HARBENS (VISCOSE SILK MANUFACTURERS.), LTD. (B.P. 285,958, 20.11.26).—In the manufacture of viscose filaments by the process in which a small quantity of a coagulating agent, *e.g.*, solutions of acids or salts, is added to the viscose before spinning, improved results are obtained if a predetermined proportion of the coagulating solution is pumped into and intimately mixed with the viscose as the latter passes to the spinning nozzle. The mixing device may be jacketed to facilitate temperature control. Suitable apparatus is described.

D. J. NORMAN.

Treatment of cellulosic material. F. C. NIEDERHAUSER and H. B. KLINE, ASSRS. to INDUSTRIAL RAYON CORP. (U.S.P. 1,661,493, 6.3.28. Appl., 18.12.26).—An alkaline solution of an alkali zincate is incorporated with viscose solution prior to the ripening operation.

D. J. NORMAN.

Treatment of cellulosic material. F. C. NIEDERHAUSER and H. B. KLINE, ASSRS. to INDUSTRIAL RAYON CORP. (U.S.P. 1,661,574, 6.3.28. Appl., 20.9.27).—To obtain viscose threads which dye evenly, a zinc salt is added to the usual sulphuric acid–sodium sulphate setting bath in such quantity that the liquid contains not more than 0.9% Zn.

A. R. POWELL.

Manufacture of laminated fabric. **Treatment of cellulose fabrics.** D. DE GÖNCZ and A. S. JONES, ASSRS. to ARNOLD PRINT WORKS (U.S.P. 1,661,880—1, 6.3.28. Appl., [A] 19.2.25, [B] 9.12.26).—(A) Webs of cellulose fibre may be joined together by treating them with a caustic alkali, immersing them in a solution containing a cuprammonium compound, and bringing them together prior to the completion of the reaction. (B) Natural cellulose fibre fabrics are impregnated with a solution of a caustic alkali, dried, and subjected to the superficial action of a solution containing a cuprammonium compound.

A. R. POWELL.

Treatment [weighting] of materials made with or containing cellulose derivatives. BRIT. CELANESE, LTD., H. DREYFUS, and G. H. ELLIS (B.P. 285,941, 23.10.26. Addn. to B.P. 281,084; B., 1928, 227).—Improved results are obtained if the solution of the loading metal contains thiocyanate in a proportion not exceeding that which is chemically equivalent to the loading metal present. Thus the loading solution may contain per litre 280 g. of stannic chloride ($\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$) and 243, 182, or 121 g. of ammonium thiocyanate, which quantities are approximately those required for the

formation of the salts $\text{Sn}(\text{CNS})_4$, $\text{Sn}(\text{CNS})_3\text{Cl}$, and $\text{Sn}(\text{CNS})_2\text{Cl}_2$, respectively.

D. J. NORMAN.

Manufacture of carbohydrate derivatives. W. HARRISON (B.P. 286,331—2, 30.10.26).—Xanthates of cellulose, starch, or other carbohydrates are treated cold or warm (*e.g.*, 60°) with (A) ammonia or its inorganic derivatives, such as hydroxylamine, (B) with organic derivatives of ammonia containing at least one unsubstituted hydrogen atom attached to nitrogen, such as primary and secondary amines, carbamide, cyanamide, guanidine, acetamide, etc., in presence of an oxidising agent (ferricyanide, hypochlorite, air with a catalyst, electrolytic nascent oxygen, etc.) which converts the xanthate into the more reactive dioxanthogen compound. The products are not ammonium salts, but are of the type, $\text{RN}:\text{C}(\text{OR}')\cdot\text{S}_2\cdot\text{C}(\text{OR}'):\text{NR}$, or, in the case of arylamines at the higher temperatures, ethers of 1-hydroxy-

benzthiazole, $\text{Ar} \begin{array}{c} \diagup \text{N} \\ \diagdown \text{S} \end{array} \text{C} \cdot \text{OR}'$, R' being the carbohydrate residue.

C. HOLLINS.

Manufacture of structural insulating boards of exploded lignocellulose fibre. W. H. MASON, ASSR. to MASON FIBRE CO. (U.S.P. 1,663,503—4, 20.3.28. Appl., [A] 19.6.25, [B] 18.9.25).—(A) Lignocellulose is exploded at pressures above 275 lb./in.² The fibrous product is converted into sheet form in hot water, passed through press rolls, and dried. (B) Lignocellulose fibre is exploded under high pressure and dried at elevated temperature and pressure. The product contains the greater part of the lignins of the original material and is porous throughout, but its exterior is less porous, stronger, and harder than the interior.

F. G. CLARKE.

Manufacture of powdered plastic material. Soc. CHIM. DES USINES DU RHÔNE (B.P. 275,558, 12.4.27. Fr., 3.8.26).—Finely-powdered fillers, colouring agents, and plasticising agents are suspended in a solution of a cellulose ester or ether and the whole is precipitated by, *e.g.*, the addition of water. The cellulose ester or ether solvent should be without solvent action on the fillers etc. The product is separated, washed, and dried, and can be moulded by heat and pressure.

D. J. NORMAN.

Freeing cellulose fibres from vegetable material. W. I. CHIDESTER (U.S.P. 1,659,809, 21.2.28. Appl., 11.1.27).—The material is cooked under pressure in a digester and then blown through a conduit into a tank containing a liquid at a lower temperature and pressure. The conduit should extend to a considerable distance below the surface of the liquid in the discharge tank, so that the fibrous material becomes disintegrated as it leaves the conduit.

D. J. NORMAN.

Treatment of fibrous material, *e.g.*, for the manufacture of paper. S. D. WELLS (B.P. 285,277, 20.4.27).—Fibrous material, *e.g.*, wood, is opened up, without breaking or cutting the ultimate fibres to any serious extent, by passing the softened material through a roller mill. Softening may be effected by mild digestion under such conditions that the intercellular cementitious substances are softened, but not completely dissolved: *e.g.*, loblolly pine chips may be impregnated

under 100 lb. hydrostatic pressure for about $\frac{1}{2}$ hr. with a liquor containing, per 100 pts. of dry wood, 10 pts. of sodium sulphite and 2 pts. of caustic soda; after removing the excess of liquor the chips are digested at 160° for about 2 hrs. The resulting "lignocellulose" pulps contain 63–91% of cellulose and 32–8% of lignin, and are obtained in yields of 60–85% depending on the type of wood and the severity of the softening treatment. Cereal straws may be softened in the roller mill itself by admitting hot water or steam and sodium carbonate or milk of lime.

D. J. NORMAN.

Treatment of waste liquors of paper manufacture for the recovery of caustic alkali therein. W. and E. BARRS (B.P. 285,933, 18 and 28.10.26 and 14.7.27).—The liquor is passed through concentration towers where it is exposed in a relatively finely-divided state to a counter-current of a hot gaseous evaporant, preferably waste flue gases. The moisture-saturated but still hot gases leaving the tower are further utilised to heat, *e.g.*, atmospheric air for use in the process. The concentrated liquor from the tower is fed direct to liquid-fuel burners and burnt to ash, with or without the aid of supplemental fuel, the hot gases from this process being used in the evaporating tower. In order to reduce the tendency of the ash to sinter in the incinerator, a stream of air is directed on to the flame from the burner to effect sudden cooling of the gaseous suspension of the products of combustion. A complete soda recovery plant embodying these principles is described.

D. J. NORMAN.

Centrifugal apparatus for spinning artificial silk. A. H. RAILING and H. J. ELEY (B.P. 286,927, 15.12.26).

Spinning boxes for spinning artificial silk. J. BRANDWOOD and T. W. HOLT (B.P. 287,183, 13.9.26).

Straining pulp for papermaking etc. D. RUSSELL and J. R. HAPPER (B.P. 286,814, 28.12.26).

Bleaching [wood pulp] (U.S.P. 1,662,951).—See VI. **Cellulose ester solvents** (B.P. 257,258).—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Bleaching [of fibres]. C. TAYLOR (B.P. 286,567, 12.10.27).—Cotton, artificial silk, or other vegetable material in yarn of fabric form is circulated in a liquor containing an alkali carbonate and hypochlorites or chlorine gas (preferably with addition of borax and ammonia), and, when saturated, is stored for a period up to 12 hrs., after which it is washed and, if desired, soured. When necessary, the cycle of operations may be repeated. Fibres so treated are less deleteriously affected, and are subsequently more readily dyed to an even shade.

D. J. NORMAN.

Bleaching of animal and vegetable materials. A. O. BRAGG, Assr. to INTERNAT. BLEACHING CORP. (U.S.P. 1,662,951, 20.3.28. Appl., 5.9.25).—The material, *e.g.*, ground wood pulp, is circulated in an aqueous solution containing a bisulphite, free sulphurous acid, and another acid, and is subsequently washed with water.

D. J. NORMAN.

Dyeing and mordanting of materials made of or containing cellulose derivatives. H. DREYFUS (B.P. 285,948, 12.11.26).—Cellulose esters or ethers are treated with swelling agents (formic, acetic, glycollic, or lactic acid, alcohol, acetone, "diacetone alcohol," diacetin, mono- and poly-hydric phenols, thiocyanates, cyanates, thiocarbimides, carbimides, carbamide, urethanes, thiocarbamides, thiourethanes, guanidines) before mordanting and dyeing with a mordant dye.

C. HOLLINS.

Dyeing, printing, or stencilling of materials made of or containing cellulose esters or ethers. H. DREYFUS (B.P. 285,942, [A] 23.10.26; 285,968—9, [B, C] 23, and 25.11.26).—(A) Cellulose esters or ethers are treated with a swelling agent (formic, acetic, glycollic, or lactic acid, alcohol, acetone, "diacetone alcohol," diacetin, mono- or poly-hydric phenols, thiocyanates, etc.) and then dyed with an aqueous dispersion of an insoluble coloured compound which has been previously treated with a dispersing agent (*e.g.*, sulphuricinoic acid or salts); diazotisation and development on the pretreated fibre is also included. (B) Hydroxylated *N*-alkyl derivatives of coloured compounds, other than azo dyes and containing no carboxyl group in the alkyl substituent are used for the dyeing etc. of cellulose esters or ethers. The methods of earlier patents (*e.g.*, using sulphuricinoic acid as solubilising agent) may be employed, or sufficient hydroxyl groups may be introduced to render the compound water-soluble. Examples are: 1-amino-4- β -hydroxyethylaminoanthraquinone (bluish-violet), 1- β -hydroxyethylaminoanthraquinone (scarlet), 1:4-bis- β -hydroxyethylaminoanthraquinone (blue), 1- β -hydroxypropylaminoanthraquinone (bluish-red); 1-anthraquinonylhydrazones of hydroxyacetone and glycolaldehyde (yellow to brown); 3:3'-bis- β - γ -dihydroxypropylaminoindanthrone (blue), bis- β -hydroxyethylbenzanthrone (yellow), condensation product from anthrapyrimidine and ethylene chlorohydrin (yellow); 1:1'-bis- β -hydroxyethylindigo (from *N*- β -hydroxyethylanthranilic acid condensed with chloroacetic acid and fused with alkali; blue), 5:5'-bis- β -hydroxyethylaminoindigo (blue); the product from Acridine Yellow G and galactose (greenish-yellow); the Nile Blue derivative obtained from nitrosodiethyl-*m*-aminophenol and β -hydroxyethyl- α -naphthylamine (greenish-blue). These compounds, except where otherwise indicated, are prepared from chloro- or bromo-derivatives and β -aminoethyl alcohol, or from amino-derivatives and ethylene oxide or propylene oxide. (C) The compounds used here are hydroxylated alkyl ethers or thioethers obtained from coloured phenols or thiophenols (other than azo compounds) by the action of suitable chlorohydrins, alkylene oxides, or acetone. Examples are: 1- β -hydroxyethylthiolanthraquinone (yellow), 1- β - γ -dihydroxypropylthiolanthraquinone (yellow), 1-methylamino-4- β - γ -dihydroxypropylthiolanthraquinone (reddish-orange), 1-acetamido-4- β -hydroxyethoxyanthraquinone (golden-yellow), 1- β - γ -dihydroxypropylthiolanthrapyridone (from chloroanthrapyridone and β - γ -dihydroxypropyl mercaptan; yellow).

C. HOLLINS.

Discharge printing of dyed acetate silk. O. Y. IMRAY. From Soc. CHEM. IND. IN BASLE (B.P. 285,973,

23.11. and 7.12.26).—Zinc formaldehydesulphoxylate, with or without an acid (tartaric acid) or alkali (potassium carbonate), together with the usual thickening agents is used for printing white discharges on acetate silk dyed with azo colours. Addition of a suitable dye to the printing paste produces a coloured discharge.

C. HOLLINS.

Hair dye. W. KRITCHEVSKY (U.S.P. 1,663,202, 20.3.28. Appl., 29.3.26).—A shampoo-dye consists of soap, glycol, and *p*-phenylenediamine. Suitable compositions may consist of soap, solvents, and homocyclic bases having three substituents which may be a hydroxy- or an amino-group, or either of these groups further substituted, two of the nuclear substituents being in positions capable of giving a quinonoid structure.

F. G. CLARKE.

Treatment of fabrics. HEBERLEIN & Co. A.-G. (B.P. 284,686, 4.10.27. Ger., 4.2.27. Addn. to B.P. 276,352; B., 1928, 260).—The wool-like effect obtained by the prior process is enhanced if the material is treated with caustic soda solution of mercerising strength at a raised temperature, *e.g.*, at 40° for 5 min. This treatment may be preceded or followed by a treatment with caustic alkali solution at the ordinary temperature.

D. J. NORMAN.

Production of pattern effects on textile goods. HEBERLEIN & Co., A.-G. (B.P. 268,389, 28.3.27. Ger., 27.3.26).—Textile material composed of or containing cellulosic fibres is passed between embossing rollers heated above 100°, and is then subjected to the action of a mercerising solution. Those parts of the fabric which have been exposed to heat and pressure are less affected by the solution than is the remainder of the fabric.

D. J. NORMAN.

Spot-dyeing of yarn. Dyeing of yarn. ECLIPSE TEXTILE DEVICES, LTD., Assees. of [A] J. P. GAREY, and [B] J. P. GAREY and L. P. HASBROUCK (B.P. 269,146—7, 11.3.27. U.S., 8.4.26).

Continuously scalding and dyeing cotton in bundles or waste. J. ANNICQ (B.P. 287,011, 27.10.27).

Machine for dyeing or similarly treating fabrics with liquids. J. BAILEY and W. H. WADSWORTH (B.P. 287,233, 17.12.26).

Treating materials made with cellulose derivatives (B.P. 283,941).—See V. **Sulphonation of fatty acids** (B.P. 263,117).—See XII. **Coloration of lacquers** (B.P. 275,969).—See XIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Examination of sulphuric acid for selenium. R. C. WELLS (J. Washington Acad. Sci., 1928, 18, 127—128).—0.5—1.0 g. of potassium bromide is dissolved in 3—4 c.c. of bromine water and distilled with 100 g. of sulphuric acid from a retort. The distillate is collected in 3—4 c.c. of concentrated hydrochloric acid saturated with sulphur dioxide and cooled. Selenium bromide comes over as a yellow liquid which deposits red selenium in contact with sulphur dioxide. As little as 1 pt. in 10⁷ of selenium can be detected. The method is preferred to the colour test with aspidospermi-

ne, which gives variable coloration according to acid strength and time of heating.

C. IRWIN.

Synthesis of ammonia under high pressure. F. MÜLLER (Arch. Eisenhüttenw., 1927—8, 1, 517—523; Stahl u. Eisen, 1928, 48, 405—406).—A review, with brief descriptions of the Haber-Bosch, Claude, Casale, and Mont Cenis methods of synthesising ammonia under high pressure. The possibility of recovering hydrogen from coke-oven gas by treating it with liquid nitrogen is discussed.

A. R. POWELL.

Effect of concentration and temperature on the germicidal efficiency of sodium hydroxide. M. LEVINE, J. H. BUCHANAN, and G. LEASE (Iowa State Coll. J. Sci., 1927, 1, 379—394).—The experiments were conducted with an organism isolated from spoiled ginger ale in simulation of the conditions experienced in automatic bottle-washing machines. The germicidal effect increased with length of time of exposure. (Cf. B., 1928, 70, 246.)

CHEMICAL ABSTRACTS.

Coloration of and free acid in coke-oven ammonium sulphate. I. E. SCHRAMM. II. A. WEINDEL. III. E. SCHRAMM (Brennstoff-Chem., 1928, 9, 46—47, 48, 48—49; cf. Windel, B., 1927, 363).—I. The impurities which cause the coloration of coke-oven ammonium sulphate consist principally of metallic sulphides; metallic compounds containing pyridine, cyanogen, and amines are also present. They are formed in the pipes leading to the saturator, and are conveyed into the latter by the liquor which condenses in the pipes. The effects of impurities in the sulphuric acid used and of the presence of phenols on the colour of the salt are of minor importance. Attempts to prepare an acid-free salt by sprinkling the mass with ammonia solution while in the centrifuge give a dark-coloured product owing to the precipitation of sulphides by the ammonium sulphide present in the crude ammonia. Addition of solid soda or ammonium carbonate produces a salt with an odour of ammonia and pyridine. Neutralisation of the excess acid is best carried out by pure gaseous ammonia. II. Schramm's conclusions are briefly discussed and criticised. III. A reply.

A. B. MANNING.

Conversion of methane into hydrogen and carbon monoxide. FISCHER and TROPSCH. **Ammonium polythionate.** TERRER and OVERDICK.—See II.

PATENTS.

Manufacture of sulphuric acid. H. PETERSEN (B.P. 267,885, 25.11.26).—In the manufacture of sulphuric acid wherein use is made of only one production tower, one denitrating tower, and nitrogen oxide absorption towers, the acid leaving the production tower is subdivided so that the portion for commerce is passed to the denitrating tower, while the excess is passed through the absorption towers and back to the production tower. Part or all of the excess acid may be sent first to the denitrating tower and then to the absorption towers, or part of the acid leaving the production tower and the absorption towers may be re-circulated.

W. G. CAREY.

Production of molybdic and tungstic acids from ores. METALLWERK PLANSEE G.M.B.H., Assees. of

DEUTS. GLÜHFADENFABR. R. KURTZ & P. SCHWARTZ-KOPF G.M.B.H. (B.P. 269,947, 26.4.27. Ger., 26.4.26).—Wholly or partly roasted molybdenite is melted in an inclined rotating crucible, and, after vapours of sulphur compounds cease to be evolved, the temperature is raised to 1000° and a rapid current of air is blown over the surface of the molten oxide, thereby causing rapid volatilisation. The fumes are led into a large glass-lined tank in which pure molybdenum trioxide in a dense form suitable for immediate reduction to metal is deposited. Tungsten trioxide may be purified in a similar way, but a much higher temperature is required.

A. R. POWELL.

Production of soluble alkali hydroxides. A. F. MEYERHOFER, Assee. of M. BUCHNER (B.P. 271,440, 3.5.27. Ger., 19.5.26).—Soluble alkali fluorides are converted into hydroxides by treatment with magnesia or an alkaline-earth oxide which has been converted into hydroxide by treatment with steam under pressure and subsequently wind-screened.

A. R. POWELL.

Manufacture of potassium carbonate and other potassium salts. J. H. BRÉGEAT (B.P. 286,172, 22.9.27).—Potassium chloride (75 pts.) is heated together with 130 pts. of quartz sand and 10 pts. of wood charcoal to obtain potassium silicate, the solution of which in water is decomposed by treatment with a current of carbon dioxide. The precipitated silica may be separated and used again in the process. The potassium carbonate in the solution is recovered by evaporation or is converted into other potassium salts by treatment with the corresponding acid or by double decomposition with a solution of the calcium salt.

A. R. POWELL.

Treatment of calcium cyanide. P. W. GRIFFITH, Assr. to AMER. CYANAMID Co. (U.S.P. 1,663,125, 20.3.28. Appl., 24.2.26).—The content of cyanogen in crude calcium cyanide is increased by agitating the compound with excess of liquid hydrocyanic acid, and distilling off the uncombined acid.

H. ROYAL-DAWSON.

Separation of cyanides from mixtures. G. H. BUCHANAN, Assr. to AMER. CYANAMID Co. (U.S.P. 1,660,667, 28.2.28. Appl., 8.7.24).—Sodium cyanide is separated from a mixture of the cyanide with sodium chloride by evaporating a solution of the mixture until about 60% of the chloride is precipitated, removing the chloride, and again evaporating until about 40% more of the chloride is precipitated, separating the chloride, and finally evaporating the mother-liquor.

C. O. HARVEY.

Concentration of barytes. W. O. BORCHERT, Assr. to NEW JERSEY ZINC Co. (U.S.P. 1,662,633—4, 13.3.28. Appl., [A] 27.8.24, [B] 19.10.27).—Barytes is recovered from material containing it by froth flotation using a selective flotation agent containing (A) soap or (B) cotton-seed oil pitch.

A. R. POWELL.

Utilisation of titanium materials containing iron. TITAN Co. A/S. (B.P. 275,580, 2.7.27. Norw., 3.8.26).—A mixture of ilmenite and a compound of magnesium, the alkalis, or alkaline earths is heated together with a reducing substance or in a reducing atmosphere to such a temperature that nearly the whole

of the iron is reduced to metal without appreciably reducing the titania. The product is ground and leached with 74% sulphuric acid, which dissolves the titanium compounds, leaving a residue from which iron may be recovered by magnetic separation. By carrying out the reduction in an atmosphere of nitrogen, nitrides are formed which are decomposed by the acid with the production of ammonium sulphate.

A. R. POWELL.

Manufacture of lead tetra-alkyl. (A) K. P. MONROE and (B) K. P. MONROE and K. WILLIAMS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,661,809—1,661,810, 6.3.28. Appl., [A] 25.3.24, [B] 15.10.25).—(A) An alloy of 1 at.-pt. of lead with 2 at.-pts. of an alkali metal is caused to react with an alkyl chloride in the presence of less than 5.1 mol.-% of a neutral hydroxylic compound soluble in the alkyl chloride. (B) An alloy of lead and an alkali metal is treated with an alkyl halide and an alcoholic solution of potassium hydroxide.

A. R. POWELL.

Manufacture of lead tetra-ethyl. W. S. CALCOTT, A. E. PARMELEE, and F. B. LORRIMAN, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,664,021, 27.3.28. Appl., 3.11.26).—Lead, intimately mixed with sodium and potassium, is treated with ethyl chloride.

H. ROYAL-DAWSON.

Manufacture of bismuth tartrates. P. A. KOBER, Assr. to G. D. SEARLE & Co. (U.S.P. 1,663,201, 20.3.28. Appl., 28.5.26).—A basic bismuth compound is treated at normal temperature or below with an alkali and tartaric acid.

F. G. CLARKE.

Production of alumina. METALLBANK & METALLURGISCHE GES. A.-G., Assecs. of G. SCHOENBERG (B.P. 279,870, 26.10.27. Ger., 26.10.26).—Crude alumina, substantially free from titania and silica but containing iron oxide, is obtained by treating aluminous materials with mineral acids, separating the insoluble material, and treating the solution with a basic substance, or evaporating it to dryness and heating the residuc. The calcined precipitate is purified by fusion in an electric furnace with less than 3% of its weight of sulphur or a sulphide, or in a current of gases containing sulphur compounds.

A. R. POWELL.

Recovery of bromine. J. P. ANDREW, Assr. to GEN. MOTORS CORP. (U.S.P. 1,662,355, 13.3.28. Appl., 22.10.25).—A solution containing bromide is treated with a slight excess of chlorine, and the liberated bromine is precipitated as an insoluble compound by the addition of a suitable reagent.

A. R. POWELL.

Powder adapted to liberate iodine. K. VAN ALLEN (U.S.P. 1,661,640, 6.3.28. Appl., 24.4.26).—A mixture of iodic acid, a reducing agent, and a filler is claimed.

A. R. POWELL.

Production of hydrofluoric acid from substances containing fluorine and silicon. M. BUCHNER, Assr. to A. F. MEYERHOFER (U.S.P. 1,664,348, 27.3.28. Appl., 18.6.25. Ger., 2.6.24).—See B.P. 234,852; B., 1925, 670.

Oxidation of ammonia by means of oxygen. F. G. LILJENROTH (U.S.P. 1,663,914, 27.3.28. Appl., 13.6.27. Swed., 19.8.26).—See B.P. 276,295; B., 1927, 907.

Treatment of marine algæ (B.P. 275,998).—See II. Caustic alkali recovery (B.P. 285,933).—See V. Ozone (B.P. 277,637 and 277,651).—See XI. Refining of crystals (B.P. 280,152).—See XVII. Nitrogen from vinasses etc. (B.P. 277,932).—See XVIII.

VIII.—GLASS; CERAMICS.

Influence of carbon dioxide under pressure on glass. O. K. BOTVINKIN (J. Russ. Phys. Chem. Soc., 1928, 60, 221—228).—Experiments were carried out with glass containing 50.84% SiO_2 , 3.12% Al_2O_3 , 0.20% Fe_2O_3 , 27.94% PbO , 1.40% CaO , 0.82% MgO , 15.19% Na_2O , 1.53% K_2O . The reaction takes place according to the equations (1) $\text{Na}_2\text{SiO}_3 + 2\text{H}_2\text{O} = 2\text{NaOH} + \text{H}_2\text{SiO}_3$; (2) $2\text{NaOH} + \text{CO}_2 = \text{Na}_2\text{CO}_3 + \text{H}_2\text{O}$. It was impossible to calculate the velocity coefficient as it decreased steadily owing to diffusion. The temperature coefficient of the reaction of decomposition of glass and a constant showing the relationship between the degree of decomposition and the pressure of carbon dioxide were obtained. The degree of decomposition was found to be proportional to the surface area. 10% of the total alkalis of the glass were dissolved in the cases of maximum decomposition. A. RATCLIFFE.

Porosity and permeability. PHILIPP.—See I.

PATENTS.

Manufacture of mirrors. G. F. and W. H. COLBERT (U.S.P. 1,662,564—5, 13.3.28. Appl., [A] 30.7.26, [B] 3.5.27).—A light-absorbing, glare-free mirror is obtained by treating a clean glass surface with a freshly prepared mixture of solutions of (A) a lead salt, sodium hydroxide, and thiourea, or (B) a salt of a heavy metal, ethyl alcohol, and an alkali thiourea. In a short time the glass becomes coated with a uniform, lustrous deposit of metal sulphide. A. R. POWELL.

Pottery kiln. A. C. WARD (U.S.P. 1,664,142, 27.3.28. Appl., 4.5.27).—The kiln consists of a floor heated from below, the door being supported by tiers defining tortuous passages in between, with flues between the tiers and the floor. H. ROYAL-DAWSON.

Apparatus for producing clay products. M. LANG (U.S.P. 1,663,309, 20.3.28. Appl., 11.4.24).—An arched kiln with an open bottom is provided with an endless conveyor close to the bottom, and with a series of adjustable burners at intervals along the kiln. L. A. COLES.

Manufacture of refractory articles [crucibles]. A. F. HOTTINGER, Assr. to CHICAGO CRUCIBLE CO. (U.S.P. 1,663,660, 27.3.28. Appl., 8.12.24).—A mixture of carbonaceous matter, clay, and a flux is coated with a composition which is fusible near the ignition temperature of the carbonaceous matter, and contains also a constituent fusible at the vitrifying temperature of the clay. H. ROYAL-DAWSON.

Heat-treatment of alumina and other [refractory] materials. T. S. CURTIS, Assr. to PACIFIC-SOUTHWEST TRUST & SAVINGS BANK (U.S.P. 1,662,739, 13.3.28. Appl., 7.4.25).—Alumina and other materials having a high m.p. are rendered denser and more refractory by making them into small rough shapes and causing a mixture of a combustible gas and air to burn within the pores of the mass on the principle of surface combustion. A. R. POWELL.

IX.—BUILDING MATERIALS.

Modern Portland cement plant. H. GUTTERIDGE (Proc. Inst. Mech. Eng., 1927, [4], 781—826).—A comprehensive account is given of cement manufacture, comprising raw materials, flow sheets of the wet and dry processes, descriptions of kilns, coolers, grinding, storing, and packing plants. W. G. CAREY.

Destruction of concrete and ferroconcrete [in gas works]; its origin, prevention, and cure. ORTHAUS (Gas- u. Wasserfach, 1928, 71, 145—156).—The compounds having the greatest destructive effect on concrete in gas works are sulphur dioxide, carbon dioxide, sulphates, and ammonium salts. In contact with Portland cement sulphur compounds in a gaseous form or in solution react with the free lime, and eventually convert it into calcium sulphate, the crystallisation of which in the concrete mass causes it to swell and crack; in the case of ferroconcrete the cracks allow corrosive media, both liquids and gases, to come into contact with the iron, which is thereby rapidly destroyed. Water containing soluble sulphates reacts with the calcium aluminate present to form calcium sulphaaluminate, which has an even greater expansion than gypsum. Water saturated with carbon dioxide dissolves the free lime as calcium hydrogen carbonate and renders the cement porous; ammonium salts have a similar action. For gas works and chemical works it is recommended that the concrete used should be made with a cement free from free lime and rich in alumina, e.g., containing 35—45% CaO , 5—10% SiO_2 , 35—55% Al_2O_3 , 5—15% Fe_2O_3 , and 2—3% TiO_2 . With this cement basalt or a well-graded chamotte should be used as ballast. Further protection is afforded by coating the concrete immediately after setting by spraying it with a thick viscous liquid, e.g., by the Contex process. A. R. POWELL.

Hydration of cements. II. Microscopical study of the hydration of the rapid-hardening cements. T. YOSHIOKA and K. KUMAGAE (J. Soc. Chem. Ind. Japan, 1927, 30, 671—678).—Hydration of commercial aluminous cements and super-cements of Portland cement type has been microscopically studied, using Keiserman and Blumenthal's staining method. Y. TOMODA.

PATENTS.

Manufacture of asphalt concrete. N. V. KONINKLIJKE STEARINE KAARSENFABR. GOUDA (B.P. 286,552, 29.8.27. Holl., 2.8.27).—Coarse (5—10 mm. diam.) and fine (0—2 mm. diam.) crushed stone are mixed separately with bituminous emulsion to which protein has been added to reduce the rate of coagulation, and the mixtures are then rolled together, or, alternatively, the coarse stone is mixed with the emulsion, and the fine stone is then worked into the mixture. L. A. COLES.

Mixing quick-setting cementitious materials. G. M. THOMSON, Assr. to PENNSYLVANIA GYPSUM CO. (U.S.P. 1,660,242, 21.2.28. Appl., 16.2.27).—The time of setting is controlled by regulation of the rate of agitation during mixing. B. M. VENABLES.

Manufacture of building material. INTERNAT. COPPERCLAD CO., Assees. of T. ROBINSON (B.P. 278,691, 22.9.27. U.S., 5.10.26).—A product suitable for building purposes, e.g., in the form of shingles, comprises a non-

metallic core of hardened plastic material, *e.g.*, crushed slate, sand, or other finely-divided solid mixed with an asphalt binder, and a jacket of fibrous material, *e.g.*, water-proofed coarse paper. A. B. MANNING.

Building material. R. P. PERRY, Assr. to BARRETT Co. (U.S.P. 1,663,095, 20.3.28. Appl., 10.7.20).—A mixture of a fibrous substance with an aqueous emulsion of a bituminous substance and an emulsifying agent is formed into sheets, dried, and waterproofed by treatment with a suitable mixture having a larger proportion of volatile constituents than the bituminous substance. A. R. POWELL.

Manufacture of paving material. F. MORTON (B.P. 286,949, 5.7.27).—A mixture of 80–95% of heated granite chippings and 20–5% of molten blast-furnace or coal-tar pitch is claimed. H. ROYAL-DAWSON.

Production of cellular building materials. G. M. THOMSON, Assr. to PENNSYLVANIA GYPSUM Co. (U.S.P. [A] 1,660,243, [B] 1,660,280, [C] 1,660,402, [A, B] 21.2.28, [C] 28.2.28. Appl., [A] 18.3.27, [B, C] 10.8.26).—A slurry is mixed in one chamber and passes to another chamber in which subdivided air or inert gas is blown through it from a perforated false bottom. The normal setting properties of the material may be maintained or the water of the slurry may be "colloidised." B. M. VENABLES.

Manufacture of plastic sheets or slabs [resembling tiles]. NEWTILE CORP., Assees. of N. E. NEWMAN (B.P. 267,150, 5.3.27. U.S., 5.3.26).—Slabs formed of a mixture of asbestos with cement as a binder are moulded and coated with nitrocellulose. H. ROYAL-DAWSON.

Manufacture of bricks, blocks, slabs, artificial stone, fuel briquettes, ovoids, etc. A. R. DAVIES, W. K. HUGHES, and A. G. MORGAN (B.P. 286,334, 1.11.26).—The treatment with carbon dioxide at different stages of the manufacture of moulded articles from lime and gravel etc. is effected in a closed circuit, and the residual gas is returned to the generator or stored for further use. L. A. COLES.

Impregnation of timber. J. T. HUGHES-JONES (B.P. 285,699, 18.5.27).—A naturally-occurring oil containing sulphur (*e.g.*, Kimmeridge shale oil) in an emulsified form is used, to which has been added manganese, cobalt, or nickel resinate, or a lead oxide etc., to accelerate polymerisation or hardening of the oil coating on exposure to air. B. FULLMAN.

Manufacture of bitumen-pitch type emulsions. L. KIRSCHBRAUN (B.P. 286,844, 31.1.27).—See U.S.P. 1,615,303; B., 1927, 301.

Apparatus for drying, dyeing, fireproofing, or otherwise treating timber. C. GOODALL (B.P. 286,833, 18.5.27).

Furnaces and kilns (B.P. 271,889).—See I. **Insulating boards** (U.S.P. 1,663,503—4).—See V.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Direct production of steel by means of methane. D. PERIETZEANU (Anal. min. Romania, 1927, No. 6—8).—The process eliminates the use of the blast furnace, and replaces the coke by methane. Methane at 900°

is passed, at the lowest possible pressure, through the fused metal at 1600–1700°, the process being carried out continuously in a rotatory mixer of the Wellman or Talbot type. The quantity of metal must be such that its temperature does not fall by more than 100°; after carburisation, fresh ore or scrap steel is added, and the crude steel is refined. CHEMICAL ABSTRACTS.

Technical and economic factors in the dressing of spathic iron ore from the San Fernando mine. W. LUYKEN and E. BIERBRAUER (Arch. Eisenhüttenw., 1927—8, 1, 487—482; Stahl u. Eisen, 1928, 48, 262—263).—The ore as mined contains 32.7% Fe, 6.7% Mn, 0.33% Cu, and 13.9% of insoluble matter, the copper being present as cupriferous pyrites. After crushing by stages to 22 mm., the clean ore is removed by hand-picking and the fines are further crushed and treated on jigs, tables, and magnetic separators. The iron product thus obtained contains 35.2% Fe, 7.2% Mn, 0.25% Cu, and 7.5% of insoluble matter; the recovery of iron and manganese exceeds 95%. The recovery of copper from the intermediate products is about 23%. A. R. POWELL.

Recrystallisation of transformer steel. M. VON MOOS, P. OBERHOFFER, and W. OERTEL (Stahl u. Eisen, 1928, 48, 393—403).—Recrystallisation diagrams of a transformer steel containing 4.07% Si, 0.05% C, 0.08% Mn, 0.007% P, and 0.006% S have been obtained for annealing periods of 1 hr. and 4 hrs. Recrystallisation commences at 500° when the metal has been subject to a 20% deformation by compression, and at 800° after a deformation of 5%. The new crystals begin to grow along the boundaries of the old grains and along the slip planes, but the latter seem to be the determining factor in the recrystallisation; after hot deformation of coarse-grained metal, owing to the almost complete absence of slip planes, very little recrystallisation takes place during annealing. With transformer steel of the above composition, there seems to be no critical amount of cold work required to induce recrystallisation. A. R. POWELL.

Resistance to corrosion, and mechanical properties of grey iron containing a small proportion of copper. P. B. MIKAILOV (Viestn. Metallspr., 1926, No. 9—10, 5—21).—Addition of copper (1.3%) does not reduce the strength, density, or homogeneity of the iron, but reduces its brittleness; 0.7% or more increases the hardness. Addition of 0.5—1.7% increases resistance to corrosion by sulphuric, hydrochloric, or acetic acid, or (in presence of bronze) sodium chloride solution, but not by alkalis, nitric acid, or water. Copper dissolves completely in grey iron; it does not affect the structure of grey iron with a high silicon content, but hinders the formation of cementite when the silicon content is low. CHEMICAL ABSTRACTS.

Thermal expansion of alloys of the stainless iron type. P. HEDNERT and W. T. SWEENEY (U.S. Bur. Stand., Sci. Paper 570, 1928, 22, 639—647).—The curves showing the expansion up to 1000° of nine samples of stainless iron containing 11.9—16.4% Cr and 0.09—0.13% C are all approximately straight lines up to the transformation point at 750—850°, where a sharp contraction takes place, followed by a further expansion, the curve for which is nearly parallel to but below that

extending from 0° to the transformation point. An increase in the chromium content tends to raise the transformation temperature and to decrease the contraction that then takes place. The average coefficients of thermal expansion $\times 10^{-6}$ for the samples tested over various temperature ranges were as follows:—20–100°, 10.0; 20–300°, 10.8; 20–500°, 11.6; 20–700°, 12.3; and 20–800°, 12.5. A. R. POWELL.

Characteristics of low-carbon manganese steel.

V. N. KRIVOBOK, B. M. LARSEN, W. B. SKINKLE, and W. C. MASTERS (Amer. Inst. Min. Met. Eng. Tech. Pub., 1927, No. 24, 1–30).—Experiments with steels containing up to 0.35% C and 1.4–3.96% Mn showed that the manganese lowered the transformation temperatures and retarded the rate of the changes. Quenching and drawing develop the best mechanical properties. Preliminary experiments on impact and fatigue showed better results for steels containing 1.75% Mn than for those of normal manganese content. The microstructures are discussed. CHEMICAL ABSTRACTS.

Analysis of acid-resisting bronze and antimonial lead. II. R. F. SMITH (Pulp and Paper Mag., 1928, 26, 389–390; cf. B., 1928, 197).—For the determination of tin and antimony in Babbitt metal, 0.5 g. is dissolved in 10 c.c. of concentrated sulphuric acid, 5 c.c. of water, and 20 c.c. of hydrochloric acid (d 1.18) are added, and the solution is boiled for 15 min. in a 500-c.c. conical flask carrying a thermometer which should read 105–110° about 2 in. above the surface of the liquid. After cooling and diluting with 130 c.c. of water, the antimony is titrated with permanganate. The solution is treated with 6 c.c. of sulphuric acid and 60 c.c. of hydrochloric acid, and the tin reduced by boiling with soft iron wire and titrated with iodine in the usual manner.

A. R. POWELL.

Effect of small additions of tin and cadmium on the properties of lead. J. COURNOT (Compt. rend., 1928, 186, 867–869).—The addition of up to 3% of tin and/or cadmium to lead does not affect its corrosion by concentrated sulphuric acid, whilst concentrated hydrochloric acid and 30% nitric acid have less effect than on pure lead if cadmium or tin alone is used. Cadmium alone increases the resistance to ammonium sulphide, and produces an elevation of mechanical resistance and elasticity. It also produces a decrease in malleability and an increase in oxidisability, both of which are checked by the addition of tin. The properties of the alloys are tabulated for varying proportions of these metals. Antimony does not enter into solid solution in the lead.

J. GRANT.

Nephelometric determination of small quantities of lead in presence of zinc by means of potassium chromate. L. S. VAN DER VLUGT (Chem. Weekblad, 1928, 25, 194–196).—The method is based on the comparison of the opalescence given with potassium chromate by the solution to be examined with that given by a standard dilute solution of lead nitrate, the acidity being so adjusted that no precipitation of zinc ensues.

S. I. LEVY.

Working-up [and analysis] of gold and silver scrap. W. ADOLPHI (Chem.-Ztg., 1928, 52, 109–110).

Coke for blast furnaces. MOTT. **Corrosion in gas mains.** SMITH.—See II.

PATENTS.

Manufacture of pure iron. A. MITTASCH, C. MÜLLER, and W. SCHUBARDT, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,663,916, 27.3.28. Appl., 12.5.26. Ger., 10.6.25).—Iron obtained by decomposition of iron carbonyl and contaminated with carbon and oxygen is purified by heating at 500° or over in an atmosphere free from oxidising gases. F. G. CROSSE.

Welding of cast iron. H. V. WILLE (U.S.P. 1,660,246, 21.2.28. Appl., 31.7.22).—The weld area is first coated with a metal which will not harden under welding conditions and then welded with ferrous metal containing sufficient carbon to leave free graphite in the fused metal. B. M. VENABLES.

Ferrous alloy. C. MEIER (U.S.P. 1,662,158, 13.3.28. Appl., 20.6.25).—An iron alloy suitable for casting into steel moulds contains 2–4% C, 2.5% Si, 1–3% Ti, 0.5–1% Al, 0.5–3% Mn, 0.25–1% P, and 1–3% of 60% ferrochromium. A. R. POWELL.

Manufacture of a steel alloy [for permanent magnets]. E. PAKULLA, Assr. to DEUTS. EDELSTAHLWERKE A.-G. (U.S.P. 1,661,907, 6.3.28. Appl., 18.9.24. Ger., 9.6.23).—The alloy contains chromium and at least one of the elements, tungsten, molybdenum, or cobalt; it is heated to a temperature above lowering temperature and quenched in a mild, non-aqueous hardening fluid. A. R. POWELL.

Heat-treatment of alloys. K. J. JACOBI, Assr. to PACIFIC COAST BORAX CO. (U.S.P. 1,664,175, 27.3.28. Appl., 9.6.26).—Alloy steels are subjected to a fused bath of an alkaline-earth borate. F. G. CROSSE.

Manufacture of alloys. A. CORRADINI, and Soc. METALLURGICA G. CORRADINI (B.P. 287,369, 1.7.27).—The alloy, suitable to replace bronze, consists of 50–60% Cu, 40–30% Zn, 2–6% Mn, 2–6% Ni, 0.25–1.50 Fe, and 0.25–1.50% Pb. F. G. CROSSE.

Copper-beryllium alloys and their [heat] treatment. SIEMENS & HALSKE A.-G. (B.P. 271,454, 9.5.27. Ger., 21.5.26).—Copper alloys containing up to 12% Be together with not more than 10% Ni and/or quantities of tin, aluminium, or zinc within the β -solid solution range are quenched from temperatures above 580°, and subsequently tempered at 300–500° in order to obtain great strength and hardness. In order to work the alloys they are annealed below 580° and allowed to cool slowly. A. R. POWELL.

Manufacture of beryllium or its alloys by electrolysis of fused salts. SIEMENS & HALSKE A.-G. (B.P. 278,723, 6.10.27. Ger., 7.10.26).—An electrolytic bath for the production of compact beryllium comprises a mixture of barium, calcium, and/or alkali fluorides with beryllium oxyfluoride. The bath should contain 7–8% Be, and is operated at about 1300°, the beryllium being deposited on a water-cooled cathode. The vapours evolved from the operation are collected in an electrostatic precipitation plant and returned, together with further quantities of beryllium oxyfluoride, to the bath as required. To deposit beryllium alloys, a salt of the

other metal is added to the bath or a layer of the metal to be alloyed may first be produced electrolytically on the cathode and the beryllium then deposited on this layer.

A. R. POWELL.

Alloys for turbine blades and machine parts exposed to similar conditions. HERÆUS VACUUM-SCHMELZE A.-G., and W. ROHN (B.P. 286,367, 3.12.26).—A suitable alloy consists of 10–40% Cr, 40–85% Ni, 1–40% Fe, and practically no carbon. Other constituents may be up to 15% W, up to 12% Mo, up to 6% Al, 2–20% Co, or a combination of these elements.

C. A. KING.

Production of aluminium or aluminium alloys from alumina. METALLBANK & METALLURGISCHE GES. A.-G. (B.P. 277,640, 26.8.27. Ger., 18.9.26).—The slow solubility of crystallised alumina in the electrolytic bath for the production of aluminium is overcome by quenching the alumina while molten in water.

C. A. KING.

Purification of magnesium and high-percentage magnesium alloys. I. G. FARBENIND. A.-G. (B.P. 280,530, 1.11.27. Ger., 13.11.26. Addn. to B.P. 182,948; B., 1922, 715).—The molten metal is treated at 700° with 0.5% of calcium and stirred under the refining salt layer claimed in B.P. 219,287 (B., 1925, 75) until most of the impurities have been removed and only about 0.1% Ca remains in the metal. After skimming, a further quantity of the refining salt is added followed by sufficient manganese chloride to produce an alloy containing 1% Mn; the mass is stirred at 900° until the alloy contains a maximum of 0.4% Mn, the flux removed, and a further quantity added, and finally the metal is cooled to casting temperature, skimmed, and poured.

A. R. POWELL.

Manufacture of a magnesium alloy. W. R. VEAZEY, Assr. to DOW CHEMICAL CO. (U.S.P. 1,663,963, 27.3.28. Appl., 8.12.24).—Magnesium is alloyed with 0.15% V.

F. G. CROSSE.

Refining of light metals, e.g., magnesium, aluminium, and their alloys, and recovering such metals from scrap. A. BECK, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,661,526, 6.3.28. Appl., 4.4.27. Ger., 15.1.26).—The metal to be refined is melted and stirred under a molten flux consisting of calcium chloride and fluoride in such proportions that the eutectic mixture, 82% CaCl_2 , 18% CaF_2 , is avoided.

A. R. POWELL.

Purification of metallic antimony. C. A. ROSE and C. L. READ, Assrs. to AMER. SMELTING & REFINING CO. (U.S.P. 1,662,439, 13.3.28. Appl., 24.8.26).—Crude antimony is treated with chlorine at a temperature just above the m.p., whereby the impurities are converted into chlorides, which are removed from the surface of the metal and treated for the recovery of any valuable constituents.

A. R. POWELL.

Apparatus for smelting the metallic dust in and recovering the heat from blast-furnace gases. J. M. SCHUTZ, Assr. to C. G. HAWLEY (U.S.P. 1,662,939, 20.3.28. Appl., 5.2.24).—The apparatus comprises a bottom bowl in combination with superimposed multiple tuyère rings to which high-pressure air and gas are supplied and above which is an overhanging annular abutment.

A. R. POWELL.

Extraction of tin from ores etc. H. L. SULMAN and H. F. K. PICARD (B.P. 286,795, 13.12.26; cf. B.P. 276,743, B., 1927, 847).—Fine tin ores or concentrates are formed into briquettes with a carbonaceous reducing agent and a binder, and the briquettes are heated in a current of hydrogen chloride in an inclined retort at such a temperature (610–630°) that the tin is volatilised as stannous chloride, substantially free from chlorides of other metals. When evolution of fumes has practically ceased, the hydrogen chloride is replaced by a current of steam, which serves to decompose other chlorides formed in the retort, with the regeneration of hydrogen chloride for further use, and to volatilise a further small quantity of stannous chloride. The alternating treatment with hydrogen chloride and steam may be repeated as often as is necessary for the almost complete removal of the tin from the charge.

A. R. POWELL.

Treatment of refractory ores containing precious metals. G. W. B. EVANS, Assr. to EVANS ORE REDUCTION CO. (U.S.P. 1,664,067, 27.3.28. Appl., 13.8.26).—The ores are mixed with lime, salt, and ammonium chloride, and the agitated mixture is heated in an atmosphere of hydrogen, whereby the precious metals can be separated.

F. G. CROSSE.

Sintering of zinc ores. H. J. STEHLI (U.S.P. 1,661,813, 6.3.28. Appl., 9.10.26).—Zinc sulphide ores are roasted until the sulphur content is reduced below that at which sintering can be carried out, the product is mixed with sufficient retort residues to furnish carbon for heating the mixture and reducing the sulphates present, and the mixture is subjected to blast-roasting in an oxidising atmosphere to decompose the sulphates and sinter the mass.

A. R. POWELL.

Dissociation of zirconium ores. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (B.P. 282,023, 2.11.27. Ger., 13.12.26).—The finely-powdered ore is mixed with 1–1.5 pts. of calcium carbonate and about 10% of calcium fluoride or chloride and the mixture is heated at 1200–1300° in a revolving furnace; a lower temperature may be used if steam is introduced into the furnace. After this treatment 95% of the zirconia is soluble in acid.

A. R. POWELL.

Manufacture of an abrasive-resisting metal. L. J. BARTON (U.S.P. 1,662,357, 13.3.28. Appl., 1.9.26).—The alloy comprises iron with 0.8–1.3% C, 0.3–0.6 Si, 0.6–1% Mn, and less than 0.08% (S + P). It is prepared by melting steel scrap in an acid-lined electric furnace, adding carbonaceous material, sand, lime, and cast iron, pig iron, or ferromanganese until the slag becomes yellowish-green in colour and the gases are removed from the metal, deoxidising with 0.8–1.5% of ferromanganese, adding 0.3–0.6% of crushed ferro-silicon, and finally dropping in small pieces of aluminium as the alloy is poured into moulds.

A. R. POWELL.

Coating [for metallic] moulds. H. M. WILLIAMS, Assr. to GEN. MOTORS RES. CORP. (U.S.P. 1,662,354, 13.3.28. Appl., 10.1.25).—A mould dressing comprising a finely-divided refractory material combined with a suitable bond is applied in a thin coating to the surface of the moulds, which are then heated.

A. R. POWELL.

Surface-treatment of metal articles. M. FOURMENT (B.P. 262,439, 1.12.26. Fr., 5.12.25).—In order to coat the surface of a metal article with another metal which imparts hardness or corrosion-resisting properties, the article is heated in a powder containing the coating metal in a finely-divided form, the heating being carried out in a high-frequency induction furnace.

A. R. POWELL.

Manufacture of [metallic] catalysts. I. G. FARBENIND. A.-G. (B.P. 286,123, 6.5.27. Addn. to B.P. 281,218; B., 1928, 235).—Finely-divided metallic catalysts are obtained by displacing the metal from a solution of one of its salts by treatment with a more electropositive metal or with hydrogen under pressure in the presence of an organic base or an acid amide. When hydrogen is used a small amount of the finely-divided metal to be deposited may be added as an accelerator.

A. R. POWELL.

[Electric] bright-annealing furnaces. SIEMENS-SCHUCKERTWERKE G.M.B.H. (B.P. 262,444, 2.12.26. Ger., 3.12.25).—The protecting hood rests on a packing or sealing ring composed of an easily fusible mixture of sand and salts. A mixture with sand of cuprous chloride (66%) and calcium chloride or sodium chloride (34%) is specified. The ring is preferably heated electrically.

H. HOLMES.

Method of galvanising. J. BLACKBURN and A. ALBRECHT, Assrs. to J. BLACKBURN (U.S.P. 1,663,037, 20.3.28. Appl., 7.12.25).—A bundle of articles is pickled, dipped into a bath of molten metal, and heated in a flame on removal from the bath. After separation of the articles from one another by jarring, the bundle is cooled by continuous passage through oil and into water.

J. S. G. THOMAS.

Electrodeposition of chromium. SIEMENS & HALSKE A.-G., Assees. of C. G. FINK (B.P. 275,223, 23.7.27. U.S., 27.7.26).—To obtain bright deposits of chromium plating a current density of 0.5–1.5 amp./in.² is required, and this can conveniently be applied only to relatively small surfaces. Hence to plate large surfaces only a portion of the surface is plated at a time, although the whole surface is immersed in the electrolyte and behaves as a cathode; the high current density required for depositing the chromium on the article is obtained by using a small travelling anode working from the top to the bottom of the bath so that, while chromium is being deposited on those parts of the cathode in close proximity to the anode, the remaining parts of the cathode are kept bright by the evolution of hydrogen.

A. R. POWELL.

Production of electrolytic deposits of metals or alloys. L. MELLERSH-JACKSON. From SIEMENS & HALSKE A.-G. (B.P. 286,457, 8.3.27).—Chromium, nickel, tungsten, molybdenum, and alloys of these metals with one another or with silicon or boron are obtained by electrolysis of fused alkali borates, cyanides, sulphates, or silicates in which the oxide of the metal is dissolved. Thus, for the deposition of chromium on iron the electrolyte comprises a mixture of 20 pts. of borax, 40 pts. of magnesium metaborate, 20 pts. of sodium metaborate, and 15 pts. of chromic oxide, or a mixture of 1000 pts. of potassium cyanide and 120 pts. of chromic oxide. In

the latter case by the addition of sodium silicate a deposit of chromium-silicon alloy may be obtained.

A. R. POWELL.

Preventing scorification of fire-bars, grate surfaces, etc. R. HOPFELT, Assr. to METALLISATOR BERLIN A.-G. (U.S.P. 1,663,944, 27.3.28. Appl., 29.1.25. Ger., 28.2.24).—See G.P. 410,158; B., 1925, 639.

Process and apparatus for casting metal into moulds. KRAFTMETALL AKTIEBOLAGET, Assees. of R. KOLB (B.P. 282,047, 3.12.27. Ger., 7.12.26).

Washing of minerals (B.P. 285,987).—See I. Molybdcic and tungstic acids (B.P. 269,947).—See VII.

XI.—ELECTROTECHNICS.

Power factor in high-frequency spark induction furnaces. R. DUFOUR (J. Phys. Radium, 1927, [vi], 8, 508–521).—A study of the schematic function of the furnace is made, based on certain hypotheses which enable a mathematical treatment of the subject to be developed, from which the time of charge of the condensers, the number of charges per sec., the intensities of the current and heat liberated at the end of the charge or discharge, and eventually the power factors are calculated successively. The variation of the power factor with the tension of the spark, and the relation of the number of wave-trains per sec. to the phase-angle, are also derived.

J. GRANT.

Transformer steel. VON MOOS and others.—See X. **Measurement of colour.** SANDERA.—See XVII.

PATENTS.

Electrical furnaces. Electrical heating apparatus. AMER. RESISTOR CORP., Assees. of H. N. SHAW (B.P. 257,949 and 257,950, 3.9.26. U.S., 3.9.25).—(A) A unitary resistance heating element is supported by a butt-ended engagement of terminal members with its ends. The ends of the element extend into apertures in the furnace walls so that there is a free space between the peripheries of the ends and the aperture walls. (B) The terminal or mounting is free to expand when heated, without causing appreciable deterioration of the electrical contact between the heating element and the terminal or mounting.

J. S. G. THOMAS.

Generation of ozone. SIEMENS & HALSKE A.-G. (B.P. 276,637 and 277,651, [A] 15.7.27, [B] 8.9.27. Ger., [A] 26.8.26, [B] 14.9.26).—(A) High-frequency current derived from a generator circuit is tuned to the working frequency, the secondary of a coreless transformer being arranged with the ozone generator in an oscillation circuit which is tuned to the frequency of the current fed to the primary of the transformer. (B) Inert, pulverulent or granular insulating material; e.g., glass beads, is filled into the space between the two discharge surfaces of the ozoniser, so that development of a brush discharge is prevented.

J. S. G. THOMAS.

[Electrolyte for] secondary battery or accumulator. E. A. SALAZAR, Assr. to MANUEL CONTE DE MIERES (U.S.P. 1,662,866, 20.3.28. Appl., 14.12.25).—A solution of zinc chloride is mixed with another halogen salt of zinc having less heat of formation than the chloride.

J. S. G. THOMAS.

Filament for incandescence lamps. G. W. MEISTER, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,663,560, 27.3.28. Appl., 20.2.26).—A solid solution of tungsten and a small proportion of molybdenum is claimed, the solid solution having a lower vapour pressure than tungsten at the same temperature. J. S. G. THOMAS.

[Treatment of] refractory metal filaments. M. N. RICH, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,663,564, 27.3.28. Appl., 30.10.25).—The surface of the metal is cleaned and hydrogen eliminated after swaging; the wire is then formed into filaments which are again treated for removal of impurities introduced during this process before being reduced to their final size. J. S. G. THOMAS.

[Clean-up material for] electron-discharge device. G. D. O'NEILL, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,663,561, 27.3.28. Appl., 3.3.27).—A mixture of a refractory metal and aluminium is claimed.

J. S. G. THOMAS.

Electron-emitting [refractory] material. C. V. IREDELL, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,663,553, 27.3.28. Appl., 24.2.27).—From 1 to 3% of thorium oxide and 0.5–4% of cerium oxide (based on the thorium oxide content) are incorporated with tungsten.

J. S. G. THOMAS.

Activation of refractory material [thoriated metal]. W. B. GERO, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,663,547, 27.3.28. Appl., 17.7.23).—The material is treated in an atmosphere which is reducing with respect to thoria before being drawn to final size.

J. S. G. THOMAS.

Production of thin electric insulating layers having a high resistance to leakage. A. JOFFÉ (B.P. 287,039, 8.9.26).—The surface of a dielectric which reacts with oxygen is treated with an oxidising medium. Thus cobalt or manganese siccative is added to the surface of linseed oil varnish which has been polymerised, and the surface treated with oxygen at 200°. Layers of the oil, so treated, 0.001 mm. thick will withstand electric fields of strength up to 10⁸ volts/cm.

J. S. G. THOMAS.

Impregnation of insulating materials. SOC. ITAL. PIRELLI (B.P. 276,985, 25.8.27. Italy, 31.8.26).—Air is expelled from the dried insulating material by a gas, e.g., carbon dioxide, propane, acetylene, which is very soluble in the impregnating insulating compound used, e.g., oil, after which the gas is expelled and the material impregnated with the insulating compound from which gas has been driven off.

J. S. G. THOMAS.

Heat-treatment of loaded conductors. W. E. BEATTY. From WESTERN ELECTRIC Co., INC. (B.P. 286,767, 6.12.26).—A conductor loaded with magnetic material, e.g., with an alloy containing 3% Cr, 78.5% Ni, and 8.5% Fe, is heated above the magnetic transition temperature of the loading material, e.g., 900°, and subsequently cooled in stages down to about 300° at a rate considerably slower than would be the case if the conductor were cooled freely in air. The conductor is then cooled to air temperature. The treatment increases the magnetic permeability of the alloy.

J. S. G. THOMAS.

Electric furnace. I. RENNERFELT (U.S.P. 1,664,132, 27.3.28. Appl., 18.8.26. Swed., 1.9.25).—See B.P. 276,823; B., 1927, 850.

Electric incandescence lamps [for use in light-houses]. GEN. ELECTRIC Co., LTD., and C. HIGGINS (B.P. 287,299, 21.2.27).

Electrodes for electric batteries. COMP. FRANÇ. POUR L'EXPLOIT. DES PROC. THOMSON-HOUSTON (B.P. 271,034, 29.12.26. Fr., 17.5.26).

[Closure-plates for] electric accumulators. G. H. TROTTER (B.P. 286,863, 19.2.27).

Hydrometer (B.P. 284,916).—See I. Artificial silk (B.P. 286,086).—See V. Steel alloy (U.S.P. 1,661,907). Beryllium and its alloys (B.P. 278,723). Annealing furnaces (B.P. 262,444). Chromium (B.P. 275,223). Electrodeposition of metals or alloys (B.P. 286,457).—See X.

XII.—FATS; OILS; WAXES.

Relation between the properties of soaps and their compositions. I. Soaps of stearic and oleic acids.

II. Surface tension and emulsifying power of stearic, oleic, and lauric acid soaps. M. HIROSE (J. Soc. Chem. Ind. Japan, 1927, 30, 734–42, 742–746).—I. The drop number (with Hillyer's stalagmometer), the surface tension (with du Nouy's apparatus), the viscosity (with Ostwald's viscosimeter), the lathering power (with the modified Stiepel's flask), and the washing power (compared by using indigo-lanolin cloth) of the mixed soaps of sodium stearate and oleate in various proportions have been measured. Among the samples tested the mixed soap of equal parts of stearate and oleate was the best in quality.

II. Among the mixed soaps of varying composition of sodium stearate, oleate, and laurate, the greatest lowering of the surface tension of water was observed in the case of stearic acid-oleic acid soaps; the presence of sodium laurate generally diminished this lowering.

Y. TOMODA.

Formation of iso-acids during the hydrogenation of fatty oils. IV. Presence of iso-acids in hardened plaice oil. S. UENO and Z. OKAMURA (J. Soc. Chem. Ind. Japan, 1927, 30, 817–819).—Isomerides of acids of the oleic series were found in the samples of hardened plaice oil prepared under various conditions. The amount of the iso-acids formed increases with the temperature and time of hydrogenation. Y. TOMODA.

Effect of various compounds on rate of development of rancidity in fats and oils. W. J. HUSA and L. M. HUSA (J. Amer. Pharm. Assoc., 1928, 17, 243–247).—The rate of development of rancidity in oil of sweet almonds is reduced by the addition of quinol, which also reduces the rate in lard about 50%. The following compounds have no effect on the rate: salicylic acid, acetylsalicylic acid, β -naphthol, liquefied phenol, *dl*-alanine, pyrogallol, and resorcinol. All the above substances were added in amounts of 0.5% of the fat, and rancidity was detected by the Kreis test and by the odour. E. H. SHARPLES.

Exploitation of whales. J. LUND (Chem.-Ztg., 1928, 52, 241–242).—The distribution of oil in the

blubber, tongue, entrails, bones, and flesh is described, and methods of extraction are discussed. S. I. LEVY.

Determination of sulphur dioxide in fatty substances. A. W. KNAPP and R. J. PHILLIPS (Analyst, 1928, 53, 149).—When rancid fats may be present in samples being examined for sulphur dioxide, unless a rough sorting test only is required, the gravimetric process should be used, since three 25 g. samples of very rancid cows' butter gave absorption figures equivalent to 29 pts. of sulphur dioxide per million by the volumetric process, but no trace by the gravimetric process. Moderately fresh cacao butter (25 g.) and cows' butter showed, respectively, 10 and 6 p.p.m. and an 8 months' old full-cream milk powder 19 p.p.m.

D. G. HEWER.

Calamary oil of Hokkaido. M. TSUJIMOTO and K. KIMURA (J. Soc. Chem. Ind. Japan, 1927, 30, 865—868; cf. *ibid.*, 1924, 27, 1300).—The oil is prepared from the intestines of the calamary, *Ommastrephes sloani pacificus*, of Hokkaido. The intestines contain about 20% of oil, or the liver separately about 30%; calamary oil consists mainly of liver oil. The oil, which is liquid at 20° and has an odour like that of fish liver oils, has d_{4}^{20} 0.9300, n_D^{20} 1.4833, iodine value 179.5 (the pyridine dibromide sulphate method), acid value 18.2, saponif. value 176.9, unsaponifiable matter 4.20%, colour reaction with sulphuric acid—pale reddish-violet, colour reaction with antimony trichloride in chloroform—pale greenish-blue. The fatty acids are crystalline solids having m.p. 31° (approx.), neutralisation value 188.6, iodine value 185.2, ether-insoluble bromide 76.29%, and bromine content of ether-insoluble bromide 71.37%. By the lead salt-ether method, the fatty acids are separated into liquid acids (77.4%; n_D^{20} 1.4810, neutralisation value 182.1, and iodine value 247.7), and solid acids (m.p. 52.5°, neutralisation value 211.1, and iodine value 14.1). The highly unsaturated fatty acids (n_D^{20} 1.4958, neutralisation value 176.5, and iodine value 372.1) are obtained from the fatty acids by the lithium salt-acetone method in 41.3% yield. The methyl esters of the fatty acids on distillation under 15 mm. pressure gives fractions containing (a) palmitic acid, (b) oleic acid, (c) highly unsaturated acids. The unsaponifiable matter is an orange-yellow crystalline solid and contains vitamin-A and cholesterol (2.7% of the oil). The growth-promoting activity of vitamin-A in the oil is rather superior to that of cod-liver oil.

K. KASHIMA.

Oil from *Pinus Gerardiana*. Chilgoza oil. I. S. D. HARDIKAR (J. Indian Chem. Soc., 1928, 5, 63—67).—The kernels of this tree, indigenous to North-West India and Afghanistan, contain 33.7% of an oil which consists of glycerides of linoleic and oleic acids (over 93%) and of saturated acids (about 5%). The esters of linolenic acid are absent. The oil has d_{4}^{20} 0.9144, n_D^{20} 1.4709, acid value 3.87, saponif. value 192.4, acetyl value 4.07, iodine (Hubl) 121.3, unsaponifiable matter 0.50.

G. A. C. GOUGH.

Theory of oil bleaching; drying of oils by gas-coagulation. A. EIBNER and A. GRETH (Farben-Ztg., 1928, 33, 1595—1598).—Drying oils of the linseed and poppyseed oil groups were bleached by bubbling a slow

current of dry air through them for 5—10 days at ordinary temperatures in diffused daylight (and in the case of the easily-bleached pine-seed oils in the dark). Parallel experiments were carried out using moist air. During the bleaching no development of acidity or change in viscosity occurs, and slight rises in saponif. values are explicable by lactone formation. Although oxygen—in the presence or absence of light—is essential to the bleaching process, the changes are not chemical in the sense of formation of "blown oils." The bleached oils were sealed in glass tubes with very small air space, and stored in diffused light for 7 years, together with parallel tubes of the unbleached oils. Up to 3 years no changes were apparent in the bleached oils; perilla oil and then linseed and walnut oils began to darken from this time. Poppyseed and pine-seed oils remained practically colourless for 7 years, after which time all the tubes were opened. The phenomena observed on opening and the nature of the stored oils are discussed. Autoxidation has occurred with the linseed oil group, whilst poppyseed oil is virtually unaltered, the adsorbed oxygen not having led to "gas-coagulation." The significance of α -linolein content is stressed, the satisfactory drying and film-forming properties of linseed oil etc. being counterbalanced by the subsequent tendency to disintegrate, whereas the slow-drying poppyseed oil is more permanent. Similar experiments by other workers are recapitulated, and Auer's views are criticised in the light of the present work.

S. S. WOOLF.

Oil testing. SCHLÜTER.—See II.

PATENTS.

Production of washing agents. G. PETROV and P. SHESTAKOV (U.S.P. 1,661,620, 6.3.28. Appl., 30.12.25. Ger., 20.2.25).—A washing compound is prepared from the purified product obtained by sulphonating a mixture of aromatic hydrocarbons and highly unsaturated drying oils.

A. R. POWELL.

[Rotatable cauldrons for use in] soap-making. L. H. NELLES (B.P. 287,198, 16.11.26).

Extraction press (B.P. 262,072).—See I.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Terpineol content of the turpentine obtained in the preparation of terpin hydrate. B. SÜSSKIND (Trans. Sci. Chem.-pharm. Inst. [Moscow], 1923, 6, 29—30; Chem. Zentr., 1927, ii, 2629).—The terpineol content was 9.2%.

A. A. ELDRIDGE.

Removal of ink from vulcanite. DITMAR.—See XIV.

PATENTS.

Production of waterproof impregnating, finishing, and colour coatings. A. JEREMIAS (B.P. 286,396, 20.12.26).—Rubber, collodion, shellac, and other materials insoluble in water are emulsified by the use of soap, Turkey-red oil, or other emulsifying agent, and converted into a "water-soluble" form in which they can be used more readily than usual as waterproof coating materials. The coating is finally reconverted into the water-insoluble state by means of a fixing agent containing a metal salt, e.g., alum, aluminium acetate, etc.

S. S. WOOLF.

Coating and impregnating medium. C. ELLIS (U.S.P. 1,663,160, 20.3.28. Appl., 7.12.20).—A phenol-sulphur halide resin which is not readily soluble in any one of various specified solvents, *e.g.*, alcohols, benzene, chlorobenzene, etc., is dissolved to a clear solution in a composite solvent, including at least one of these solvents.

S. S. WOOLF.

Manufacture of lithopone. NEW JERSEY ZINC CO., Assees. of [A, B] W. C. HOOEX, and [C] F. G. BREYER and C. W. FARBER (B.P. 263,119, 263,120, and 265,550, [A, B] 2.12.26, [C] 9.12.26. U.S., [A, B] 15.12.25, [C] 2.2.26).—(A) In the precipitation of "crude pulp" in lithopone manufacture, the end-point of the reaction between the barium sulphide solution and the zinc sulphate solution is determined by the amounts of hydroxide and/or hydrosulphide radical present, the reaction being controlled by predetermined excess of and ratio between these radicals. (B) This excess and ratio are such as to control the properties of the finished lithopone, *e.g.*, oil absorption, alkalinity, strength, wetability, etc., and to lower the calcining temperature below that at which the light-resistance of the pigment deteriorates. (C) The crude lithopone is precipitated in the presence of a soluble chloride or other water-soluble compounds in amount equivalent to 0.75–1.0 g. of chlorine per litre of zinc sulphate solution (*d* 1.20 at 20°), the ratio of hydroxide to hydrosulphide radical being maintained at 65–80 : 35–20. The crude lithopone is dried to a water content of 4–10% before calcination. S. S. WOOLF.

Preparation of white titanium pigments. G. CARTERET (B.P. 274,072, 30.6.27. Fr., 12.7.26).—Titanium oxide is precipitated from a hydrochloric acid solution to which a small amount of sulphuric acid has been added, or from a sulphuric acid solution to which a small amount of hydrochloric acid has been added, by boiling in the presence of a suspended base, *e.g.*, silica, aluminium silicate, etc. The resulting paste is washed, filtered, dried, intimately mixed with sufficient weak alkali solution for neutralisation, again washed, dried, calcined, cooled, and crushed. The total acidity of the acid solution of titanium hydroxide is preferably about half that indicated by the theoretical equation of the hydrolysis of the normal salt. S. S. WOOLF.

Printing in greasy inks with gelatin printing surfaces. O. TREICHEL (U.S.P. 1,661,515, 6.3.28. Appl., 30.11.26. Ger., 18.12.25).—The gelatin is treated with a substance which reduces or delays its swelling prior to use. A. R. POWELL.

Coloration of lacquers. I. G. FARBENIND. A.-G. (B.P. 275,969, 2.8.27. Ger., 13.8.26).—Lacquers are coloured by incorporation therewith of basic dyestuffs which may also contain acid groups, together with complex acids, *e.g.*, phosphotungstic acid, phosphomolybdic acid, silicotungstic acid, etc., or salts of these acids capable of forming colour lakes with the dyestuffs. S. S. WOOLF.

Manufacture of cellulose ester compositions. I. G. FARBENIND. A.-G. (B.P. 257,258, 11.8.26. Ger., 21.8.25).—Halogeno-alkyl esters of mono- or poly-basic carboxylic acids (including carbonic but excluding acetic), of b.p. above 150°, are used as solvents for cellulose esters. B. FULLMAN.

Insulating layers (B.P. 287,039).—See XI.

XIV.—INDIA-RUBBER ; GUTTA-PERCHA.

Distribution of fillers in rubber mixing. F. S. REINER (Gummi-Ztg., 1928, 43, 1359–1360).—The ordinary operation of mixing fails to give uniform distribution of the various ingredients in rubber, and even subsequent calendaring does not effect this. Examination of the rubber insulation of a sample of commercial electric wiring also revealed irregularity of composition. Uniformity of composition was observed in rubber into which the ingredients had been introduced while the rubber was in the form of latex or of solution.

D. F. TWISS.

Removing dried ink from vulcanite fountain pens. R. DITMAR (Chem.-Ztg., 1928, 52, 123).—The greenish-grey discoloration caused by prolonged immersion in water or aqueous solutions to remove dried ink may be avoided by using a solution of sodium hyposulphite, which effects a rapid cleaning without affecting the vulcanite.

S. I. LEVY.

Power consumption in the preparation of crêpe rubber. M. SCHRÖTER and R. RIEBL (Med. Proefstat. Rubber, Buitenzorg, 1927, [22], 397–419; Arch. Rubbercultuur, 1927, 11, [8]).

PATENTS.

Manufacture of rubber goods. G. FESSEL, and TECH. CHEMICALIEN COMP. G.M.B.H. (B.P. 286,171, 21.9.27).—The tars and tar distillates from the carbonisation of lignite, coal, shale, or peat are extracted with a suitable organic solvent such as hot alcohol or with a solution of sodium hydroxide. The extracted oils, which may be freed from volatile constituents by distillation, when used in the proportion of 5–10 pts., constitute an excellent softener and preservative for rubber (100 pts.). For this purpose they can be further improved by heating between 135° and 150°, and are superior to coal-tar and pine-tar oil. D. F. TWISS.

Production of rubber goods directly from latex. DUNLOP RUBBER CO., and D. F. TWISS (B.P. 285,938, 22.10.26).—A mould of the desired shape, coated with a jelly containing a coagulant, is immersed in latex, of normal or higher concentration, to which compounding and vulcanising ingredients may have been added. A deposit of coagulum is formed, which is then dried and removed from the mould. D. F. TWISS.

Preservation of vulcanised rubber. H. W. FIEDLER (U.S.P. 1,661,887, 6.3.28. Appl., 3.9.24. Ger., 13.9.23).—A paste for the preservation of vulcanised rubber is produced by mixing glycerol, benzaldehyde, and talc into a solution of raw rubber in paraffin oil at about 140° and raising the temperature to about 180°.

D. F. TWISS.

XV.—LEATHER ; GLUE.

Batch B.14 hide powder. III. D. BURTON (J. Soc. Leather Trades Chem., 1928, 12, 20–25; cf. B., 1927, 757).—Chroming with chrome alum gives less variation in the non-tans with different batches of powder than basic chromium chloride on myrobalans, quebracho, mimosa, and blended extracts. Slightly higher results were obtained for the myrobalans extract using chrome alum instead of basic chromium chloride for chroming each batch of powder, except for B.14. Greater

differences between the non-tans given by the two methods of chroming were obtained on quebracho extracts, the powder chromed with chrome alum showing the lower result. B.14 hide powder gave a considerably greater amount of non-tars for both myrobalans and quebracho extracts with either method of chroming. Comparison of the results given by B.14 powder chromed with chrome alum and B.13 chromed with basic chromium chloride on the above extracts and crushed myrobalans shows an average difference of 0.5% (—0.6% to +0.5%). D. WOODROFFE.

PATENTS.

Preparation of depilatory substances. R. BOTSON (B.P. 285,152, 11.11.26).—An alkali sulphide, e.g., sodium sulphide, is liquefied by treatment with dry steam in a closed digester and terpinolene (1%) or similar solvent is added. The liquid is decanted, filtered, and mixed with a solution of caustic soda (*d* 1.32) in the proportion of 20–10%, filtered, concentrated, and crystallised. D. WOODROFFE.

Production of glue in flake or powder form. A. EHRENREICH (B.P. 284,593, 24.3.27. Belg., 31.1.27).—The viscous fluid prepared from the waste material of plagiostomi is evaporated *in vacuo* until it contains 70% of water only, agitated to form a frothy mass either of large cells, which are dried *in vacuo* yielding flakes, or of small cells which after being dried are powdered. D. WOODROFFE.

XVI.—AGRICULTURE.

Relations between plant growth and soil reaction as affected by fertilisers and liming. K. NEHRING (Z. Pflanz. Düng., 1928, 10A, 348–366).—The degree of exchange acidity and the p_H value of soils varies seasonally. Following much rain, acid conditions are intensified in the subsoil but reappear in the surface soil during the drier periods of the year when surface evaporation is rapid. Additions of lime in excess of the amounts calculated by the Daikuhara method do not remove all exchange acidity nor bring the p_H value of the soil to 7.0. Increased yields of barley followed the liming of acid soils, the maximum point corresponding to the complete neutralisation of the exchange acidity. Maximum yields of oats were obtained when the exchange acidity value was from 2.5 to 6.0 c.c. Decreased yields resulted from liming to a greater or less extent. On soils exhibiting exchange acidity physiologically alkaline fertilisers gave better yields of barley than acid ones, but both were equally satisfactory after adequate liming. Acid fertilisers were preferable to alkaline for oats. A. G. POLLARD.

Yield of oats as a function of the nitrogen supplied and the growth period. R. MEYER [with A. STORCK] (Z. Pflanz. Düng., 1928, 10A, 329–347).—Cropping experiments are examined from the point of view of the Mitscherlich law of growth, but with separate and detailed consideration of root, grain, and straw development, and the period of the life history of the plant. Up to the ripening stage of oats the slope of the nitrogen-growth curve does not increase with increasing phosphate treatment, but the position of the maximum point approaches that corresponding to the heavier

nitrogen treatments. The descending portion of the curve has a point of inflexion. Increasing additions of nitrogen produce growth-curve maxima first in the roots, followed by straw and grain in order. The maxima of a series of nitrogen-growth curves move towards the position of the higher nitrogen values as the growth period increases. Nitrogen-growth curves for roots reach their maxima earlier than those for the aerial portions of the plant. From a consideration of nitrogen-growth curves for varying periods a process is derived for determining final crop yields and thence fertiliser requirements. A. G. POLLARD.

Manuring of meadows with mineral nitrogen. GISEVITZ and KLITSCH (Z. Pflanz. Düng., 1928, 7B, 1–15).—Chemical examination of hay from fertilised meadows showed that potash-phosphate fertilisation produced heavier crops with higher nitrogen content than nitrogen fertilisation. Complete artificials produced the heaviest crops, but the nitrogen content was below that when potash and phosphate only were used. These results were paralleled by the proportion of grasses to clovers following the use of the various fertilisers. Where more than one cut of grass was taken in a season on plots receiving nitrogen only, the total yield and nitrogen content were higher when three cuttings were made than with two. Meadows fertilised with phosphate and potash only yielded heavier crops and higher nitrogen contents when two cuttings were made than with three. Ammonium sulphate, sodium nitrate, and urea did not exhibit very definite differences in their effects on herbage. Sodium nitrate was generally superior in dry weather. The bearing of these results on the control of pasture and meadow land is discussed. A. G. POLLARD.

Examination and manuring of acid mineral soils. H. KAPPEN (Z. Pflanz. Düng., 1928, 7B, 16–29).—Methods for measuring soil acidity are compared and discussed. To characterise soil acidity, determinations should be made of p_H value, exchange and hydrolytic acidity, and absolute neutralisation value (Kappen-Hilkowitz). The degree of saturation can be calculated from these latter values. The efficiency of liming depends on the fineness of grinding of the material, thoroughness of distribution through the soil mass, and the temperature and moisture content of the soil. The value of determinations of buffer capacity (Jensen) in the selection of general fertiliser materials is discussed. A. G. POLLARD.

Phosphate in the soil solution as affected by reaction and cation concentrations. L. J. H. TEAKLE (Soil Sci., 1928, 25, 143–162).—Experiments on the influence of various cations on the solubility of phosphates in aqueous solutions, and of various reagents on the concentration of phosphate in the soil solution indicate that a purely chemical explanation of the low phosphate concentration of the soil solution is most satisfactory. A series of reactions are proposed as indicative of the behaviour of phosphate in the soil. Iron, manganese, aluminium, and calcium are effective in turn in precipitating phosphate from the soil solution as the reaction changes from very acid to neutral and alkaline conditions. H. J. G. HINES.

Sodium salts, used in conjunction with potash, as a plant food. V. PEAS. H. HEINRICH (Z. Pflanz. Düng., 1928, 10A, 299—328).—Heavy dressings of potash manures fail to bring about corresponding crop increases in peas. The further addition of sodium salts, however, increases the yield of both grain and straw in amounts depending on the amount of salt added. A fairly large proportion of the commonly used potash fertilisers for peas may, with advantage, be replaced by sodium salts. Definite relationships between the extent of the replacement and the crop yields exist. Relatively large doses of sodium salts are without injurious effects on the crop, and in moderate amounts sodium sulphate alone produces considerable crop increases. Explanations of these phenomena are discussed. A. G. POLLARD.

Action of sodium chloride in irrigation water on certain plants. N. PASSERINI and P. GALLI (Atti R. Accad. Lincei, 1927, [vi], 6, 618—619).—The results of pot experiments indicate the inadvisability of using water containing more than 1 pt. of combined chlorine per 1000 for the irrigation of crops in open ground. This limit may be increased to 2 or even 3 pts. per 1000 in the case of the spontaneous growths of established meadowland or arable land with permeable subsoil.

T. H. POPE.

Absorption of rain water during vegetation by the soil and its utilisation by plants. N. TULAIKOV and A. KOZHEVNIKOV (Soil Sci., 1928, 25, 213—224).—Moisture determinations at intervals of 5 cm. were made to a depth of 100 cm. during spring and summer on soils bearing winter rye, spring wheat, and sunflower, and the results are discussed in relation to the rainfall and the habits of these plants.

H. J. G. HINES.

Significance of soil respiration in the carbon dioxide-feeding of plants. P. HASSE and F. KIRCHMEYER (Z. Pflanz. Düng., 1928, 10A, 257—298).—Methods are described for the determination of carbon dioxide in the free atmosphere and the extent of the daily "respiratory" changes in the soil mass. The average carbon dioxide content of the atmosphere near to the soil surface was 2·2% higher at night than during the day. The carbon dioxide concentration during soil respiration depended to a large extent on temperature and moisture conditions of the soil, and was greater at night than during the day. Exhalations of carbon dioxide from cropped soils varied with the crop, being much larger under lucerne than under potatoes or rye. 80% of the respired carbon dioxide in the case of lucerne came from the plant roots. The concentration of carbon dioxide in the free air at the zone of the middle leaves of the plants was smaller than that of the upper air during the day and greater during the night. The range of concentrations differed with the crop, being greatest with lucerne. The near presence of factory exhaust gases did not affect the carbon dioxide concentration of the atmosphere immediately surrounding the plants. The effects of air currents and local temperature changes are discussed. Changes in the carbon dioxide of the air, sufficient to produce significant crop increases, are not considered to be producible by manurial treatment. Systematic "gassing" of crops by carbon dioxide is not a commercial possibility unless waste gas can be utilised.

A. G. POLLARD.

Fixation of [atmospheric] nitrogen by *Bacterium aerogenes* and related species. C. E. SKINNER (Soil Sci., 1928, 25, 195—205).—Of twenty-three strains of *B. aerogenes* isolated from soil, flour, and water, two, possibly three, strains were found to fix atmospheric nitrogen when grown in routine nitrogen-free media. Of the strains of *B. radiobacter* examined some were unable to fix nitrogen, and no strains of *B. cloacae* were found capable of so doing.

H. J. G. HINES.

***Actinomyces acidophilus*, N.Sp.—a group of *acidophilus actinomycetes* isolated from the soil.** H. L. JENSEN (Soil Sci., 1928, 25, 225—234).—This new species-group was isolated from three acid humus soils (p_H 3·4—4·1). They are able to live only in acid media. Their biochemical activities are briefly described.

H. J. G. HINES.

Constitution and properties of certain common anti-speronospore preparations. U. PRATOLONGO and M. P. ALLAN (Giorn. Chim. Ind. Appl., 1928, 10, 3—7).—The efficacy of Bordeaux mixtures as anti-cryptogams is due to basic copper sulphates, such as the compounds, $CuSO_4 \cdot 3CuO \cdot 3H_2O$ and $CuSO_4 \cdot 4CuO \cdot 4H_2O$; that of Caffaro paste to the compound, $3CuO \cdot CuCl_2$, that of Caffaro powder to $3CuO \cdot CaCl_2$, and that of copper protector (Protector ramato) to $CuCO_3 \cdot Cu(OH)_2$. The most soluble of these is the calcium copper oxychloride, and the least soluble the copper oxychloride and basic carbonate, the resistance to washing-off being the reverse of the solubility. Poisoning of cattle by fodder (beet-tops) which has been treated with these preparations is unlikely, in view of the low solubility of the basic copper compounds in the slightly acid gastric contents.

T. H. POPE.

p_H value of some Texas soils and its relation to the incidence of certain woody plant species. A. H. BERKMAN (Soil Sci., 1928, 25, 133—142).—Colorimetric determinations of soil reaction in conjunction with ecological observations afford some evidence that soil reaction exerts a selective influence on woody plant species. This evidence is as yet regarded as inconclusive.

H. J. G. HINES.

Chemical characteristics of tobaccos of various districts (1925 season). A. SCHMUCK and BALABUCHA-PORZOWA (State Inst. Tobacco Invest., U.S.S.R., Bull. 40, 1927, 41 pp.).—Results are given of chemical analyses of tobaccos grown in 1925 in Abkhasia, Armenia, Black Sea region, Tuapsinski, Crimea, etc. In general, the proportions of carbohydrates and of polyphenolic compounds are greater, and those of proteins and total nitrogen less in the better than in the inferior tobaccos. The latter contain also the higher percentages of nicotine and ash. An indication of the quality of a tobacco is furnished by the ratio of the percentage of total carbohydrates to that of proteins, this ratio increasing as the quality improves. Of Crimean tobaccos, the paler ones are superior to the darker varieties.

T. H. POPE.

Effect of different fertilisers on the amount and quality of tobacco crops. A. V. OTRYGANIEV and D. V. BALANDA (State Inst. Tobacco Invest., U.S.S.R., Bull. 43, 1928, 33 pp.).—The results of field experiments during 1926 and 1927 on tobacco of the

Oriental cigarette type show that heavy reddish-brown silt loam soil gives a tobacco of continually improving quality and increasing yield as the amount of P_2O_5 applied to the soil as superphosphate is increased from 22 to 65 kg. per hectare. Thomas meal produces better results than superphosphate, but addition of lime with the meal lowers the yield. The use of potash and nitrogenous fertilisers alone, or of lime alone, results in no marked increase in the yield. On sandy loam soil the greatest need is phosphatic fertiliser, but the crop is increased also by nitrogen and potash, but not by lime; less pronounced improvement in yield follows the use of stable manure. The effects of ammonium sulphate and sodium nitrate are practically identical as regards the magnitude of the crop, but tobacco of better quality is obtained when ammonium sulphate is used. Organic fertilisers, especially green manure (lupin and hairy vetch), improve the quality more than the inorganic fertilisers. Only when lime was applied, either alone or with other manures, was any appreciable change in the soil reaction produced.

T. H. POPE.

Fertiliser from fermentation waste. BAUER.—See XVIII. **Ripening of Grimes apples.** PLAGGE and others.—See XIX.

PATENT.

Sulphur fungicide and insecticide. C. D. VREELAND (U.S.P. 1,662,550, 13.3.28. Appl., 24.11.25).—An intimate dry mixture of flowers of sulphur and crude pyroligneous acetate is claimed.

A. R. POWELL.

XVII.—SUGARS; STARCHES; GUMS.

Objective measurement of colour in sugar manufacture. K. ŠANDERA (Z. Zuckerind. Czechoslov., 1928, 52, 261—269).—The author has designed an instrument for measuring intensities of light by means of the photo-electric current produced at a surface of metallic potassium in a photo-electric cell. It consists of a 100-watt lamp with light filters and stops, from which the light passes through a glass cell containing the liquid under examination to a photo-electric bulb or cell containing a film of potassium and a grid anode. The current which passes between the film and the anode on illumination is amplified by a three-electrode thermionic valve and measured by means of a millivoltmeter. For light of given quality the photo-electric current is, within wide limits, proportional to the intensity of illumination. Measurements made on sugar juices at various stages of manufacture show agreement of duplicate readings to within 1 in 150. The instrument can be used to measure not only colour intensities, but also turbidities.

J. H. LANE.

Rate of dissolution of sugar. K. ŠANDERA (Z. Zuckerind. Czechoslov., 1927, 52, 153—160).—The rate at which commercial sugars dissolve in water is important to the refiner, since it is the controlling factor in affination. Experiments were made on single large crystals, and on regularly-shaped pellets of amorphous sugar, to determine the time taken for the object to dissolve completely when supported in a wire-gauze cage immersed just below the surface of water in a relatively deep vessel. The rate of dissolution (weight of crystal or pellet

divided by time required for dissolution and by original surface area) at 20° was of the order of 1.6×10^{-4} g./cm.²/sec., irrespective of the origin and shape of the sugar. Corresponding values for some inorganic compounds are: potassium hydroxide 18×10^{-4} , potassium iodide 15.3×10^{-4} , copper sulphate 0.6×10^{-4} , and potassium permanganate 0.0097×10^{-4} . Comparative experiments on the initial rate of dissolution of raw beet sugars when washed with water were made by placing 10 g. of the sugar in a receptacle with glass bottom and wire-gauze sides, and allowing a stream of water to flow on it from a certain height at such a rate that 100 c.c. of washings passed through the receptacle in 1 min. The proportion of the total sugar dissolved in the 100 c.c. of washings ranged from 35% to 74% for different raw sugars, whilst the proportion of mineral matter dissolved ranged from 68% to 86%.

J. H. LANE.

Rate of dissolution of sucrose under various physical and chemical conditions. V. NETUKA (Z. Zuckerind. Czechoslov., 1928, 52, 289—293).—Using similar apparatus to that of Šandera (cf. preceding abstract), the author investigated the rate of dissolution of pellets of amorphous sugar prepared by allowing drops of molten sugar (containing 2—3% of moisture) to fall into cold mercury. The relative rates of dissolution of these pellets in pure water at 20°, 40°, and 70° were approximately in the proportions 1:4:13. At a constant temperature of 20° the rate of dissolution in pure water was twice that in 18% sucrose solution, and about 10 times as great as the rate in 50% sucrose solutions. In lime water, 1% solutions of potassium sulphate and oxalic acid, and 5% solution of sodium carbonate, the pellets dissolved rather less rapidly than in pure water, but the maximum observed reduction in rate was only about 5%. In molasses solutions also the rate of dissolution was less than in pure water, owing mainly to the sugar and only to a slight extent to the salts present. The rate of dissolution of sugar crystals in pure water at 20° showed occasional differences beyond the limits of experimental error. It is suggested that crystals formed slowly in the cold may dissolve more slowly than those formed under the usual technical conditions.

J. H. LANE.

Determination of the polarisation of beets by aqueous digestion, and the errors due to the volume of the marc. V. STANĚK and J. VONDRÁK (Z. Zuckerind. Czechoslov., 1927, 52, 165—174).—After the polarisation of beets had been effected by the method of hot aqueous digestion, the undissolved matter was treated with successive cold water washings, each removed by centrifuging, until all the sugar was extracted. The combined liquors were then concentrated, *in vacuo*, and polarised. This check on the usual procedure confirmed previous conclusions (B., 1927, 395) that the generally accepted values for the volumes of the marc and the juice are incorrect, a conclusion which has also been confirmed by Spengler and Brendel (B., 1927, 234), and by Fremel. From the whole of their data the authors calculate that the average volume of the marc in the normal weight (26 g.) of beet is 1.54 c.c. instead of 0.6 c.c., and the average volume of the juice is 21.78 c.c. instead of 23.0 c.c., as generally accepted

at present. They recommend, therefore, that in analysing beets by aqueous digestion by the graduated flask method the flask should be marked at 403 c.c., instead of 401.2 c.c. for 52 g., and if the pipette method is used the volume to be added to 52 g. of sample should be 356.4 c.c. instead of 351 c.c. J. H. LANE.

Preparation of syrups. I. II. E. E. PETERSON, M. LEVINE, and J. H. BUCHANAN (Iowa State Coll. J. Sci., 1927, 2, 31—41, 43—55).—83.3% of 734 samples of spoiled carbonated beverages and 47% of 132 samples of commercial sugar contained yeast. The addition of sodium carbonate to sodium hydroxide wash-waters is not so effective for killing yeasts as the addition of an equivalent amount of hydroxide. Simultaneous inversion and sterilisation of sucrose syrups (40—90 g. in 100 c.c.) may be effected by adding to 100 c.c. 1 c.c. of 7.074*N*-citric or 6.553*N*-tartaric acid, and boiling for 5 min. In concentrated syrups, equilibrium is reached at 97.5% inversion. The velocity of the reaction (considered as unimolecular) increases as hydrolysis progresses, and increases slightly with increasing concentration up to 600 g. per litre.

CHEMICAL ABSTRACTS.

Bone charcoal and active vegetable carbons [in sugar refining]. C. MRASEK (Z. Zuckerind. Czechoslov., 1927, 52, 174—176).—Bone charcoal is considered to give more regular and surer working, and, contrary to Wiesner's views, it is more economical than vegetable carbons. J. H. LANE.

Decolorisation of [sugar] juices by active carbons. F. NOSEK (Z. Zuckerind. Czechoslov., 1928, 52, 269—272).—A commentary on recent papers by Wiesner (B., 1928, 103) and Mrasek (cf. preceding abstract) on the use of active carbons in refinery and factory working. J. H. LANE.

Reducing power of carbons. VAŠÁTKO.—See II.

Fertiliser from molasses waste.—BAUER.—See XVIII.

PATENTS.

Continuous defecation of sugar juices. DORR Co., Assees. of E. R. RAMSEY and A. W. BULL (B.P. 270,757, 9.5.27. U.S., 8.5.26).—The juice is treated with milk of lime followed by the regulated addition of carbon dioxide, so that the liquid is maintained at a predetermined alkalinity, which is controlled by the measurement of the electrical resistance of a portion of the solution in which the reaction is complete.

F. R. ENNOS.

Refining of massecuite or other crystalline materials. RAFFINERIE TIRLEMONTAISE SOC. ANON. (B.P. 280,152, 29.11.26. Ger., 4.11.26).—The massecuite is centrifuged in an apparatus of the usual single-drum type, which rotates at a greatly increased speed, so that the mother-liquor is completely separated from the crystals, thereby obviating the necessity of washing and drying the crystals. F. R. ENNOS.

Manufacture of starch compositions particularly suitable for use as adhesives. O. MEYER (B.P. 286,377, 6.12.26).—Starch is mixed with 2—3% of cyclohexanone or methylcyclohexanone and is then treated with a solution of sodium hydroxide. After

neutralising with an organic acid the product is dried at 50—70° and pulverised. F. R. ENNOS.

Carbohydrate derivatives (B.P. 286,331—2).—See V. **Nitrogen and acetone from molasses** (B.P. 277,932).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Composition of wines of the Malaga type. E. ŠVAGR, J. LUKAS, and A. JILEK (Chem. Listy, 1928, 22, 29—34).—Malaga wines manufactured in Czechoslovakia differ little in chemical composition and alimentary value from the original products. The calorific value of the above wines is very nearly the same as that of malt wines of the Malaga type, both ordinary and medicinal. In composition, however, the malt Malaga types differ from the true Malaga wines in their high content of lactic acid and dextrin, and in the absence of tartaric acid. Whilst unmaturing malt Malaga wines contain sucrose, this is not a specific constituent. The mineral content of the latter wines is only half as great as for true Malagas, and is remarkable for its low potassium and high phosphoric acid content. R. TRUSZKOWSKI.

Preparation of fertiliser from fermentation waste. E. BAUER (Oesterr. Chem.-Ztg., 1928, 31, 39).—The waste materials from the production of industrial alcohol from molasses contain potassium salts and most of the nitrogen from the sugar beet. Recovery of the nitrogen by burning is uneconomical as nitrogen is lost, and the preparation of a suitable fertiliser is made difficult on account of the presence of glycerin. Good results can, however, be obtained by mixing the materials with "saturation lime." The product contains 2—2.3% N, 5—5.4% K₂O, 0.6—1% P₂O₅, 28—30% CaO (as carbonate), and 26—28% of organic matter.

R. H. GRIFFITH.

PATENTS.

Production of acetone and butyl alcohol by fermentation. COMMERCIAL SOLVENTS CORP., Assees. of W. J. EDMONDS (B.P. 268,749, 14.3.27. U.S., 3.4.26).—Acetone and butyl alcohol are produced by fermentation of a carbohydrate mash in the absence of air. The entire series of operations of charging, fermenting, and removal of a suitable mash into and from the fermentation vessel are carried out in an atmosphere of "fermenter gas" which has been obtained from a previous fermentation. This gas, which consists of a mixture of carbon dioxide and hydrogen, is, prior to using, sterilised and freed from air, nitrogen, or oxygen. C. RANKEN.

Manufacture of dihydroxyacetone. I. G. FARBER-IND. A.-G. (B.P. 282,347, 21.9.27. Ger., 18.12.26. Addn. to B.P. 269,950; B., 1928, 313).—The prior process is modified by using a nutrient medium containing extractive substances from the waste products of malt, e.g., brewers' grains. C. RANKEN.

Recovery of nitrogen and acetone from vinasses and/or molasses. NOUVELLES INDUSTRIES CHIMIQUES SOC. ANON. (B.P. 277,932, 13.6.27. Belg., 21.9.26).—The vinasses and/or molasses are mixed at 100° with an excess of lime or other alkaline-earth base (over 50% by wt.) and destructively distilled below 600° and under 1 atm. in a current of inert or reducing gases,

e.g., nitrogen, carbon dioxide, hydrogen, or methane. Use is preferably made of the gases obtained from the destructive distillation after recovery of the useful by-products. The carbonaceous residues are used as decolorising agents. B. FULLMAN.

Boiling and evaporating apparatus for boilers, particularly for boilers for brewing. H. KOCK and R. QUITT (B.P. 286,955, 15.7.27).

XIX.—FOODS.

Refraction of milks with less than 8.5% of solids-not-fat. G. D. ELSDON and J. R. STUBBS (Analyst, 1928, 53, 150—151; cf. B., 1927, 375).—Continued application of the refractometric method to milks having a percentage of solids-not-fat less than 8.5 has confirmed the opinion that the determination of the refraction of the serum offers no advantage over that of solids-not-fat of the milk, and may even be less valuable. The values given, covering a year's samples, support the contention that the mixed milk of a herd seldom gives a refraction less than 37.0 with copper sulphate serum at 20°. D. G. HEWER.

"Alkalinity" of milk and its electrolytic determination. Z. VON MARIKOVSKY and E. LINDNER (Chem.-Ztg., 1928, 52, 283).—By "alkalinity" of milk is understood the total alkalinity of the cations present in the milk; it may be calculated from the analysis of the ash, or determined directly by electrolysis, using a mercury cathode and dissolving in standard acid the metals deposited in the amalgam. The cathode is prepared by cutting off the closed end of a small pyrex test-tube and covering the other end with a filter paper and a piece of clean linen held in place with a rubber band; 2 c.c. of pure mercury are placed inside the tube so as to rest on the filter paper, and a platinum wire dipping into this connects with a source of current at 30—40 volts. The cathode is placed in a small beaker containing 5 c.c. of milk diluted with about 50 c.c. of water, and a small platinum strip is used as anode. Electrolysis is continued for about 40 min., 10 c.c. of 0.1*N*-sulphuric acid being poured in the cathode tube above the mercury before switching on the current. Titration of this acid at the end of the operation yields a value for the alkalinity of the milk; the c.c. of acid used are divided by the c.c. of milk taken. For normal milks this value is approximately a constant, 1.12—1.14; higher values indicate that the milk has been neutralised. A. R. POWELL.

Pimento for colouring egg-yolks. W. A. MORGAN and J. G. WOODRUFF (Ga. Exp. Stat. Bull., 1927, 147, 210—215).—Waste pimento fed to hens (0.4 g. per day) greatly intensifies the colour of the egg yolk.

CHEMICAL ABSTRACTS.

Physical and chemical changes of Grimes apples during ripening and storage. H. H. PLAGGE, A. J. MANEY, and F. GERHARDT (Iowa Agric. Exp. Sta. Res. Bull., 1926, 91, 1—72).—A discussion of indications of the optimal harvesting time. The ripening processes of fruit on the tree and in storage are associated with loss of moisture, acidity, dextrin, starch, and material hydrolysable by acid, and an increase in density, sugars, and soluble pectin. CHEMICAL ABSTRACTS.

Report of the Preservatives Determination Committee of the Chemists of the Manufacturing Confectioners' Alliance and of the Food Manufacturers' Federation. Determination of sulphur dioxide [in foods] (Analyst, 1928, 53, 118—129).—The finely-divided sample (25—100 g.) is introduced with 200 c.c. of de-aerated water into a 500 c.c. round-bottomed flask holding a tap-funnel, still-head, and, where necessary, inlet tube for steam. The special "B.A.R." still-head recommended is described. The upper end is connected to a vertical condenser, which is fitted with an adapter of the scrubber type. The receiving beaker contains water to which has been added 0.2—0.3 c.c. of a filtered 1% starch solution and a few drops of 0.05*N*-iodine solution, or 1—5 c.c. if large quantities of sulphur dioxide are anticipated. Phosphoric acid (25 c.c. of 20% acid) is run into the distilling flask, and the liquid brought to the boil within 2½ min. If more than 10 mins.' distillation is required steam should be used. At least 90% of the sulphur dioxide comes over in the first rush of gas. The end-point may usually be taken as that at which more than 1 min. is required to decolorise 0.1 c.c. of 0.05*N*-iodine. As distillation proceeds 0.05*N*-iodine is added from a burette so that the colour remains. If a gravimetric determination is desired the distillate is filtered, made up to volume, 10 c.c. of 0.1*N*-hydrochloric acid are added, the liquid is boiled for 5 min., 2.5 c.c. of 10% barium chloride are added drop by drop, the solution is again boiled for 5 min., 2.5 c.c. more barium chloride are added, and the liquid is left to simmer for 1 hr. before cooling. It should then be kept for at least 2 hrs. before filtering. For starch a 1-litre flask should be used, 100 g. of sample taken, and hydrochloric acid used instead of phosphoric acid. Gelatin, meats, and dried fruits require steam-distillation, and every endeavour should be made to complete distillation in 10 min. for gelatin and 20—30 min. for hard fruits. If decomposed protein is likely to be present sulphur dioxide and hydrogen sulphide must be differentiated. D. G. HEWER.

Effect of other reducing substances on the determination of sulphur dioxide [in foods]. J. W. BLACK and B. J. W. WARREN (Analyst, 1928, 53, 130—132).—In determining sulphur dioxide by distillation it was found that the bulk of the gas comes over in the first few minutes after distillation has started. By rapid titration during the evolution of gas, curves may be constructed which, in the case of all substances tried in which sulphur dioxide was present, show a steep upward curve to a certain point corresponding approximately to the point where added sulphur dioxide ceases to be evolved, followed after 8—10 min. from the beginning of distillation by a change of direction. In the case of glucose syrup, gelatin, and sausage the curve is then more or less horizontal, but where iodine-reducing substances are present, as in spices, the angle is more obtuse. The angle formed between the two portions of the curve is thus an indication of the proportion of interfering substances, and where these are present (nutmeg, mustard, ginger, etc.) a time limit should be set for distillation when determining sulphur dioxide. D. G. HEWER.

Determination of sulphur dioxide in sausages and in foods by distillation in a vacuum. H. O. JONES (Analyst, 1928, 53, 138—141).—When sulphite is added to sausage there is an almost immediate loss of sulphur dioxide; e.g., on addition of 450 pts. per million there is a very approximate loss of 150 pts. Sulphur dioxide determinations in sausages 3, 24, and 48 hrs. after making gave values showing a total diminution of about a third, and in each case the Preservatives Determination Committee's method gave higher values than Leach's method. The determination of sulphur dioxide by vacuum distillation (and, in the presence of phosphoric acid, in carbon dioxide) into iodine solution gave results which agreed closely with the Committee's method in the case of sodium metabisulphite, gelatin, and sausage, and were higher than by Leach's or Monier-Williams' method, but the method is more complicated and slower than that of the Committee. D. G. HEWER.

Rapid determination of sulphites [in foods] by alkaline liberation or extraction, and titration. H. R. JENSEN (Analyst, 1928, 53, 133—135).—Direct titration with iodine after formation of fully hydrolysed sulphite by cold digestion for 15 min. with sodium or potassium hydroxide, followed by strong acidification, is recommended. *Glucose*.—The syrup (50 g.) is dissolved in 50 c.c. of water at 50°, cooled to 15°, and 20 c.c. of 5% sodium hydroxide solution are added. After 15 min. 30 c.c. of 20% sulphuric acid and 100 c.c. of water are added, and the liberated sulphur dioxide is titrated with 0.05*N*-iodine. Results, which indicate the absence of other iodine-absorbing substances for combined sulphite, vary between 22 and 90%, usually about 60%. *Sucrose*.—Commercial sugar may contain invert sugar, and the maximum value obtained by alkalisation methods may often be used as a sorting test. *Cornflour*.—A quick indication of the maximum amount of sulphur dioxide present may be obtained by adding 100 g. of starch powder to a cold mixture of 303.5 c.c. of water with 40 c.c. of 5% sodium hydroxide. After occasional rotation for 15 min., followed by filtration, a mixture of 30 c.c. of 10% hydrochloric acid with 20 c.c. of water is added to 200 c.c. of the filtrate. After again filtering at once, 200 c.c. are titrated with 0.05*N*-iodine solution. A total displacement value of 69.2 c.c. was found for 100 g. of starch, and an insoluble volume of 12.7 c.c. by double dilution. D. G. HEWER.

Sulphur dioxide in fatty substances. KNAPP and PHILLIPS.—See XII.

PATENTS.

Treatment of lactic fluids. J. M. W. KITCHEN (U.S.P. 1,658,168, 7.2.28. Appl., 19.3.23).—Before losing its self-preservative power, the clarified milk is cooled below 5°, aerated, pasteurised by heat, and again cooled to —2° to +5°, each process being performed in an atmosphere free from dust and bacteria, prior to being distributed. F. R. ENNOS.

Production of artificial coffee oil. INTERNAT. NAHRUNGS- U. GENUSSMITTEL-A.-G. (B.P. 260,960, 22.9.26. Ger., 4.11.25).—Coffee oil (cf. B.P. 246,454; B., 1926, 1028) contains hydrogen sulphide, methyl mercaptan, furfuryl mercaptan, dimethyl sulphide, acetaldehyde,

α -methyl-*n*-butaldehyde, furfuraldehyde, methylfurfuraldehyde, acetone, furyl ketone, diacetyl, pentane- α - β -dione, methyl and higher alcohols, furfuryl alcohol, acetic acid, isovaleric acid and esters, palmitic acid, phenol, pyrocatechol, guaiacol, vinylguaiacol, 2:3-dihydroxyacetophenone, pyridine, pyrazine, methylpyrazine, 1-methylpyrrole, 1-furfurylpyrrole, and naphthalene. The synthesised oil can be used as flavouring.

C. HOLLINS.

Manufacture of oleomargarine. H. LEROUDIER (U.S.P. 1,663,913, 27.3.28. Appl., 7.5.26. Fr., 20.5.25).—See B.P. 252,369; B., 1927, 503.

Production of white and purified pectin. R. PAUL and R. H. GRANDSEIGNE (B.P. 286,914, 29.4.27).—See F.P. 614,882; B., 1928, 34.

Conservation of foodstuffs for lengthy periods under chilled conditions. FOOD CHILLERS, LTD., ASSEES. OF A. R. McLEOD (B.P. 275,184, 24.6.27. Austral., 30.7.26).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

p_H determinations in alcoholic solutions. R. B. SMITH (J. Amer. Pharm. Assoc., 1928, 17, 241—243).—The p_H values of a number of alcoholic tinctures have been determined by means of an apparatus constructed from readily available materials. Stable and reproducible values are obtained, and the following U.S.P. tinctures have been measured: digitalis 5.12—5.77, aconite 5.20—5.51, strophanthus 5.43, and ergot 4.97.

E. H. SHARPLES.

Chilgoza oil. HARDIKAR.—See XII.

PATENTS.

Manufacture of new polyamino-compounds. W. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 286,087, 19.11.26. Addn. to B.P. 267,169; B., 1927, 379).—The aminoalkyl side-chains of the prior patent are modified by the introduction of ether or thioether linkings. E.g., β -(6-methoxy-8-quinolylamino)ethyl β -diethylaminoethyl ether [8- β -(β -diethylaminoethoxy)ethylamino-6-methoxyquinoline], b.p. 213—215°/0.5 mm., is prepared by heating 8-amino-6-methoxyquinoline with the β' -chloro- β -diethylaminodiethyl ether, b.p. 72—73°/5 mm., obtained from β -diethylamino- β' -hydroxydiethyl ether [glycol mono- β -diethylaminoethyl ether] and thionyl chloride. The sodium compound of the last-named ether then reacts with ethylene chlorohydrin to give β -(β -hydroxyethoxy)ethyl β -diethylaminoethyl ether [glycol β -diethylaminoethyl β -hydroxyethyl ether], $\text{CH}_2(\text{OH})\cdot\text{CH}_2\cdot\text{O}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{O}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{NEt}_2$, b.p. 132—136°/7 mm., from which by the action of thionyl chloride β -(β -chloroethoxy)ethyl β -diethylaminoethyl ether [glycol β -chloroethyl β -diethylaminoethyl ether] is obtained; this reacts with 8-amino-6-methoxyquinoline to form glycol β -(6-methoxy-8-quinolylamino)ethyl β -diethylaminoethyl ether [8- β -(β -diethylaminoethoxy)ethoxyethylamino-6-methoxyquinoline], b.p. 238—240°/2 mm. Mono-thioglycol, b.p. 67°/18 mm., obtained from ethylene chlorohydrin and sodium hydrosulphide, is condensed with β -chlorotriethylamine to form β -diethylamino- β' -hydroxydiethyl sulphide, b.p. 122°/6 mm.; the corre-

sponding β' -chloro-compound resulting from the action of thionyl chloride reacts with 8-amino-6-methoxyquinoline to give β -(6-methoxy-8-quinolylamino)- β' -diethylamino-diethyl sulphide, b.p. $240^{\circ}/2$ mm. C. HOLLINS.

Manufacture of new alkaloid salts of camphoric acid. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 269,498, 10.3.27. Ger., 15.4.26).—Alkaloids of the *Solanacea* group, particularly scopolamine, hyoscyamine, and atropine, are combined with equimolecular proportions of camphoric acid. The presence of excess of alkaloid does not affect the formation of the salts, which are useful therapeutically. C. HOLLINS.

Manufacture of new therapeutic compounds [pure ergotoxine]. WELLCOME FOUNDATION, LTD., and G. M. TIMMIS (B.P. 286,400, 23.12.26).—For the liberation of ergotoxine from the sulphate or phosphate, obtainable by the methods of Kraft (A., 1906, i, 979) and Barger and Carr (B., 1907, 483), weak alkalis, such as sodium bicarbonate or borax, are used, giving a purer alkaloid, which after crystallisation from benzene has $[\alpha]_{D}^{20} -195^{\circ}$, or -156° ($+2C_6H_6$). C. HOLLINS.

Preparation of soluble salts of ergotoxine. WELLCOME FOUNDATION, LTD., and G. M. TIMMIS (B.P. 286,582, 23.12.26).—*Ergotoxine methanesulphonate*, m.p. 214° (corr.), and *ethanesulphonate*, m.p. 209° (corr.), made from the alkaloid and the corresponding acids in alcohol, are stable and readily soluble in water. C. HOLLINS.

Manufacture of arseno-bismuth compounds. E. C. R. MARKS. From ABBOTT LABORATORIES (B.P. 286,115, 26.8.26).—3 : 3'-Diamino-4 : 4'-dihydroxyarsenobenzene, in the form of hydrochloride or formaldehyde-bisulphite compound, is stirred with an aqueous solution of potassium tribismuthyl tartrate, $C_4H_2O_9Bi_3K_4H_2O$, at 15° , to form a compound of the type, $AsAr(Bi : AsAr)_2$, which is precipitated by pouring the reaction mixture into methyl alcohol and ether. C. HOLLINS.

Preparation of 2-chloropyridine. DEUTS. GOLD- u. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 281,650, 28.11.27. Ger., 3.12.26).—2-Chloropyridine is obtained when an *N*-alkyl-2-pyridone, e.g., *N*-methyl-2-pyridone, is treated at 120° under reflux with (gaseous) phosgene. The yield is 93–94%. C. HOLLINS.

Preparation of camphene. W. SCHULENBURG, ASSR. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,663,569, 27.3.28. Appl., 26.2.27. Ger., 27.2.26).—Pinene hydrochloride is converted into camphene by elimination of hydrogen chloride by means of an alkali monoxide. B. FULLMAN.

Manufacture of hormone from the sexual organs. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 265,166, 6.1.27. Ger., 26.1.26).—Fresh, comminuted sexual organs (ovaries, placenta, etc.) are extracted with methyl alcohol at an elevated temperature below its b.p. The extract is evaporated, and the dry, water-soluble residue repeatedly extracted with methyl alcohol, evaporation of which yields a highly active oil. B. FULLMAN.

Preparation of active germinal gland substances in a water-soluble form. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 276,994, 29.8.27. Ger.,

4.9.26).—Hormone extracts, obtained from the germinal glands by, e.g., the process described in B.P. 261,356 (B., 1927, 619), are brought into aqueous solution free from salts by treatment in the presence of water with alkaline-earth hydroxides and removing the latter, after heating, with carbon dioxide. B. FULLMAN.

Preparation of a toxin specific to the pneumococcus, and of a pneumococcus antitoxin. F. B. DEHN. From E. LILLY & Co. (B.P. 286,744, 11.5.27).—Pneumococcus toxin is prepared by incubating a pure culture of pneumococci for 20–40 hrs. in a fresh broth which is made from fresh beef, sterilised under pressure, and free from added carbohydrate. The toxin is obtained organism-free by Berkefelding. It produces the symptoms of pneumonia on injection into animals, from the blood of which the corresponding antitoxin (effective in treating pneumococcal infections of man or animals) may be obtained. B. FULLMAN.

Pharmaceutical product. W. SCHOELLER, A. FELDT, M. GEHRKE, and E. BORGWARDT, ASSRS. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,663,390, 20.3.28. Appl., 1.6.26. Ger., 2.6.24).—See B.P. 234,806; B., 1925, 692.

Extraction presses (B.P. 262,072).—See I. Mercaptans (B.P. 286,152).—See III. Coffee oil (B.P. 260,960).—See XIX.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Metallic silver content of photographic films. F. WEIGERT and F. LÜHR (Naturwiss., 1927, 15, 788; Chem. Zentr., 1927, ii, 2638).—By a modification of Cox's method of differential titration, 0.001 mg. of silver ions in 20 c.c. of solution could be determined with 0.001*N*-potassium iodide solution. The films were attacked by a simple method employing nitric acid. The silver content of commercial films was $1.82\text{--}2.80 \times 10^{-4}$ mg./cm.² That this is ripening silver was shown by the determination of silver in freshly prepared and in ripened silver halide emulsions. With silver chloride the quantity of silver increased rapidly, with silver bromide more slowly, and with silver iodide not at all. Treatment of a plate with persulphate reduces the amount of silver and reduces the sensitiveness. Since silver sulphide is not dissolved by persulphate, it appears that the sensitive nuclei are not composed of silver sulphide. A. A. ELDRIDGE.

Rate of desilverisation of the wet collodion silver bath. B. P. O'SHAUGHNESSY (Phot. J., 1928, 68, 123–127).—The use of the argentometer does not give an accurate indication of the absolute amount of silver nitrate in a sensitising bath in use. A chemical method of determination must be used. The amount of silver nitrate used per unit area of plate during sensitising is dependent to some degree on the personal equation of the worker. The discrepancy between the argentometer reading and the actual amount of silver nitrate present at any given stage is greater in the dipping bath, where evaporation is at a minimum, than in the open bath. The greatest disturbance of the argentometer readings is caused by the introduction into the bath of ether and alcohol from the collodion. W. CLARK.

Colour of developed silver images. A. and L. LUMIÈRE and A. SEYEWETZ (Brit. J. Phot., 1928, 75, 172—173).—The colours of images developed with developers which insolubilise the gelatin are due to the formation of a secondary image superimposed on the reduced silver image and modifying its colour. The secondary images are probably formed of quinone compounds arising from oxidation of the developer. The colour varies with the nature of the developer and occasionally with that of the alkali used. Its intensity is determined essentially by the sulphite content of the developer, but depends also to a certain degree on the nature of the developing agent. The intensity diminishes as the proportion of sulphite is increased. With pyrogallol the secondary images fail to appear with a sulphite content of 10%; with other developers it is 2%. Glycin is the only developer not giving the oxidised image, whatever the sulphite content. The gelatin in which the oxidised product is formed is insolubilised to a degree corresponding with the intensity of the secondary image. This image is a mordant for basic dyes, and may be intensified by fixation of these dyes to such an extent as to become equal, or even superior, in density to the original silver image. W. CLARK.

Chemical effects of radiation. P. VILLARD (Phot. J., 1928, 68, 118—121).—A *résumé* of the author's recent views on the antagonistic actions of radiations, and the method of latent image formation (cf. A., 1927, 218, 323). W. CLARK.

PATENTS.

Manufacture of photographic sensitive layers. F. VAN DER GRINTEN (CHEM. FABR. L. VAN DER GRINTEN) (B.P. 281,604, 23.3.27. Holl., 11.12.26).—The sensitive layer consists of the diazo compound of an *NV*-asymmetrically disubstituted *p*-phenylenediamine or a derivative thereof (excluding such as form diazo-anhydrides), with or without a suitable alkaline-coupling component and sufficient non-volatile acid to prevent premature coupling. Diazosulphonic acids or their salts may be used, and the sensitive layer may be mixed with a suitable lake-forming material such as barium chloride. Examples are: *p*-dimethylaminodiazobenzene, with tartaric acid and barium chloride, for development with Naphthol AS (black); *p*-diethylaminodiazobenzene, with potassium dihydrogen citrate and barium chloride, for development with β -naphthol (brown-black); 2-diisamylamino-*m*-xylene-5-diazonium chlorostannate, with citric acid and 2:7-dihydroxynaphthalene, for development with alkali (dark violet). C. HOLLINS.

Production of printed transparent images. SANDOR G.M.B.H. (B.P. 265,924, 10.12.26. Ger., 11.2.26).—An absorptive, opaque layer of cellulose derivatives is coated on a smooth surface, printed, and then clarified to the required extent by a fresh application of a solvent or gelatinising agent which acts on the cellulose particles, or of a solution of a cellulose derivative or resin containing the constituents which act on the cellulose particles. If the images are required for projection, they may be detached from the support and then clarified, or else prepared on a translucent or transparent base material. W. CLARK.

Colour-screen films. R. RUTH, and AKTIEBOLAGET SVERIGES LITOGRAFISKA TRYCKERIER (B.P. 285,977, 23.11.26).—The screen elements consist of a colloid laid directly on a colloid support having the same physical and optical properties as the screen elements. They are fixed to the support by pressure and heat, the support being superficially softened on the surface receiving the screen particles which are applied in a single layer. The film is then fed between pressure rollers, the roller acting on the screen grains being heated. W. CLARK.

Colour screens for photographic purposes. J. R. ROBERTSON (B.P. 286,052, 22.1.27).—The screens consist of vacuum chambers in conjunction with optical lenses. The chambers each contain a different gas, and means are provided for producing an electric discharge in the gas in any particular chamber, or in a number of chambers in succession in the case of colour cinematography. The different gases assume different colours when the tubes are excited. W. CLARK.

Preparation of photographic developers. P. SCHESTAKOFF (U.S.P. 1,663,959, 27.3.28. Appl., 29.4.25. Ger., 12.5.24).—See F.P. 600,532; B., 1926, 566.

[Multicolour screens for] colour photography. W. CARPMAEL. From J. H. POWRIE (B.P. 276,188 and 287,388, 11.10.26).

XXII.—EXPLOSIVES; MATCHES.

PATENT.

Preparation of progressive burning smokeless powder. B. TROXLER, ASST. to HERCULES POWDER Co. (U.S.P. 1,661,278, 6.3.28. Appl., 16.11.26).—Grains of smokeless powder are coated with a deterrent substance by mixing the two together with just sufficient water to moisten the surfaces of the powder grains and heating the mixture in air to a temperature only just above the m.p. of the deterrent substance with agitation. A. R. POWELL.

XXIII.—SANITATION; WATER PURIFICATION.

Industrial hygiene in relation to the manufacture of artificial silk in Italy. U. POMILIO (Giorn. Chim. Ind. Appl., 1928, 10, 7—11).—The growth of the artificial silk industry, particularly in Italy, hygienic measures for dealing with the noxious gases emitted during the manufacture of viscose silk, and the composition and evaluation of the aqueous residues from such manufacture are considered. In one of the large Italian factories it is found that of 340 g. of carbon disulphide combined as xanthate per kg. of viscose yarn about 90 g. undergo decomposition, giving rise to 55 g. at most of hydrogen sulphide. With a daily output of 6000 kg. of the silk, the ventilators have a total capacity of 12,000 m.³/min., and the concentration of the hydrogen sulphide should be reduced to 0.02 g./m.³. Examination of the air inside and immediately outside the building at different times of the day and under different meteorological conditions showed sometimes absence of hydrogen sulphide and in other cases a minimum concentration of 0.000493 g. and a maximum of 0.008547 g. of hydrogen sulphide per m.³, the mean being 0.0035 g. At none of the various factories examined is injury to the health

of the workpeople experienced, and experiments on guinea-pigs exposed for months to the action of air containing 0.022 g. of hydrogen sulphide per m.³ fail to reveal any harmful effects on the health of the animals. The aqueous effluent, the composition of which varies considerably, contains per litre: hydrogen and other sulphides 0.9–4.9 mg., organic matter 0.0122–0.0796 g., total SO₄ 0.1311–1.7359 g., residue at 90° 0.7845–2.1765 g., and acidity (as SO₄) 0–0.2982 g. As the effluent is diluted at least 5 to 10 times in the stream into which it flows, injury to the aquatic fauna or flora is considered impossible. In a report published in May, 1925, Loriga considers no system of ventilation to be sufficient for viscose silk factories, and recommends the destruction of the deleterious products by combustion or other method of oxidation.

T. H. POPE.

Comparative results from the testing of various germicidal agents. G. F. LEONARD and E. HEACOCK (Amer. J. Pharm., 1928, 100, 103–111).—The phenol coefficients of more than a thousand samples of various germicidal agents have been tested by the Hygienic Laboratory method (Hygienic Laboratory Bulletin, 1912, No. 82) using specific cultures of *B. typhosus* (Hopkins) and *Staphylococcus aureus* (No. 73). The cultures, taken from stock, were transplanted monthly, incubated for 24 hrs., and stored in the refrigerator. Three days before making a test they were transferred to beef extract broth and daily transplants made on to bouillon. A 24-hr. broth culture was used for the test, the medium consisting of beef extract, peptone, and sodium chloride in water adjusted to pH 7; 0.1 c.c. of bouillon culture was added to 5 c.c. of germicide or phenol solution at 20° and samples were transplanted to bouillon tubes at intervals of 1, 5, 10 and 15 min. in the staphylococcus test and 5, 7½, 10, 12½, and 15 min. in the typhoid test, and the tubes incubated for 48 hrs. The phenol coefficient is the arithmetic mean of the three ratios (highest killing dilution of germicide: that of phenol) for the 5, 10, and 15 min. intervals. The cultures did not alter in their resistance to phenol during 8 years. This method of procedure gives uniform and consistent coefficients with the two organisms.

S. COFFEY.

Danger of mercury vapour. W. KRÖNER (Chem.-Ztg., 1928, 52, 121–122).—A résumé of recent publications from the Verein für Innere Medizin of Berlin, which have confirmed the great danger of poisoning to workers and others handling mercury, or even to those frequenting premises on which the metal is allowed to remain exposed.

S. I. LEVY.

Sedimentation studies of turbid American river waters. A. W. BULL and G. M. DARBY (J. Amer. Water Works' Assoc., 1928, 19, 284–305).—The comparative clarification rates of various rivers have been measured by determining the percentage reduction in suspended solids after varying periods of settlement in litre cylinders. The results obtained were found to agree fairly well with those from practical-scale operation. When deeper cylinders with a continuous or intermittent feed were used, the results for short detention periods were too low owing to a high upward velocity in the cylinder and the excessive depth of the feed tube.

The advantage of a slow stirring arrangement in densifying sludge is demonstrated, and settlement of the water prior to the addition of flocculating or softening reagents is shown to result in lower running costs. C. JEPSON.

Turbidity and coagulant dosage [of river water]. K. C. ARMSTRONG (J. Amer. Water Works' Assoc., 1928, 19, 306–312).—The proportion of coagulant required to produce a satisfactory water may be regulated by determining the turbidity after 3 hrs.' settlement. If the water is very turbid after settlement in grit chambers, the coagulant may be applied with advantage in successive doses. The turbidity after 24 hrs.' settlement indicates the amount of very finely-divided material, and must also be taken into account when fixing the amount of alum required. C. JEPSON.

Effect of slightly alkaline tap water on spawn and eggs of trout and perch. E. S. HOPKINS (J. Amer. Water Works' Assoc., 1928, 19, 313–322).—Experimental studies indicate that free carbon dioxide is necessary for the sustenance of fish life, and that a high oxygen content, even in very cold water, is not detrimental. C. JEPSON.

Testing for iodine in potable waters. P. DRAWE (Chem.-Ztg., 1928, 52, 122–123).—The total halides in 50 litres of the water are precipitated as the silver compounds in the usual way; these are collected on a filter, the silver chloride is removed by washing with ammonia, the insoluble residue treated with sulphuric acid and metallic zinc, and the iodide determined in the solution in the usual way after filtering. S. I. LEVY.

PATENTS.

Fumigant material. H. W. HOUGHTON, Assr. to SAFETY FUMIGANT Co. (U.S.P. 1,663,082, 20.3.28. Appl. 25.6.25).—Material for producing a mixture of hydrogen cyanide and cyanogen chloride in lachrymatory proportions on treatment with hydrochloric acid comprises a cyanide and an alkali chlorate worked up to a plastic mass with slaked lime, sand, and sodium chloride, and then allowed to harden. L. A. COLES.

Bactericidal preparation. P. LALAND, Assr. to NYEGAARD & Co., A./S. (U.S.P. 1,662,930, 20.3.28. Appl. 19.5.26. Norw., 15.3.26).—A bactericidal colloidal preparation comprises a combination of silver with a boroglucoside obtained from *Rhizoma Rhei*.

A. R. POWELL.

Removal of germs from water. O. & R. ADLER (B.P. 286,338, 4.11.26).—The residual chlorine, left after sterilisation and filtration through a fine porous medium such as sand, quartz powder, etc., is removed by passing the water over coarse pieces of carbon which converts the free chlorine into chlorine ions. The carbon is prepared by mixing it in a pulverised form with a binding material (e.g., clay), moulding to shape, and baking. C. JEPSON.

[Automatic] apparatus for softening or purifying water or for carrying out processes based on exchange reactions. UNITED WATER SOFTENERS, LTD., R. H. THOMSON, and H. S. LAWRENCE (B.P. 288,018, 11.3.27).

Bactericidal agents (U.S.P. 1,661,568).—See IV.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

MAY 25, 1928.

I.—GENERAL; PLANT; MACHINERY.

Heat transfer for oils and water in pipes. F. H. MORRIS and W. G. WHITMAN (Ind. Eng. Chem., 1928, 20, 234—240).—Film coefficients of heat transfer in a jacketed pipe were determined for the heating and cooling of water and more particularly of petroleum oils with a view to the rational design of pipe stills and coolers. The rate of flow was 1—20 ft./sec., flow being turbulent. The general equation for the heat-transfer coefficient, h , is $hD/k = \phi(Du\rho/z) \times \psi(cz/k)$, where D is the pipe diameter, k the conductivity, u the velocity, ρ the density, c the specific heat, and z the viscosity of the liquid; ϕ and ψ represent experimentally determined functions. The physical properties were taken at the temperature of the main body of the liquid, more concordant results being so obtained. The pipe used was long enough to minimise "end effects." The relationship between the three variables in the above equation was determined by maintaining one constant and plotting the relation between the other two graphically. Curves are thus obtained which provide data suitable for design, but the values of $\phi(Du\rho/z)$ for cooling are only about 75% of those for heating, necessitating the use of separate curves for the two cases. C. IRWIN.

Computation methods in counter-current absorption systems. W. K. LEWIS and W. H. McADAMS (Ind. Eng. Chem., 1928, 20, 253—257).—If a gas is being treated for the removal of a solute by a liquid in a column and operation is uniform $G(y - y_0) = L(x - x_0)$, when G , L are the rates of flow of gas and liquid, and y , y_0 the concentration of solute in the gas at any point and at the top, and x , x_0 concentrations in the liquid. This (linear) relationship is plotted for comparison with equilibrium curves for the systems considered (the drying of air by sulphuric acid and the scrubbing of gas for benzol). The equilibrium curve itself represents conditions at the interface between the two phases, and the rate of transfer of material is proportional to the surface of contact and the concentration gradient through each of the two films. From these facts a further series of equations is derived which are simplified if one or other film controls the transfer. These equations are integrated graphically. In the case of the system inert gas—benzene—absorbing oil Raoult's law is followed and $y = \rho x [\pi - (\rho - \pi)x]$, where ρ is the pressure of pure benzene and π the total pressure. The various diagrams are discussed in detail. C. IRWIN.

Universal tank-calibration chart. A. K. DOOLITTLE (Ind. Eng. Chem., 1928, 20, 322—323).—A chart was prepared to calibrate boiler tanks with bulged ends, whether horizontal or vertical. Two sets of curves were plotted on logarithmic paper to the equations $A =$

$\int_0^h 2x dy$; $x = \sqrt{r^2 - (y - r)^2}$, and $v = 7.4805 AL$ measurements being in inches (depth), feet (length), and gallons (volume). The abscissae common to both sets represent the area of the segmental cross-section of liquid. A further separate set of curves gives the correction for the ends, which are treated as segments of a sphere of radius equal to the tank diameter. For simplicity the volume was integrated as an ellipsoid of revolution $v = (2 - \sqrt{3})/3 \times \pi h^2(3r - h)$, and a correction of 0.767 applied to bring the volumes so obtained to the true ones. C. IRWIN.

Boiler scale and its prevention. H. WALDE (Wiss. Veröff. Siemens-Konz., 1927, 6, 151—170).—A method of presenting the results of analyses of boiler feed-waters and of boiler scales has been elaborated in order to classify the various types of incrustation and bring them into relation with the feed water. Three ratios are calculated for the water, and three for the deposit—the ratios of silica to lime and of magnesia to lime for both, the ratio of sulphate hardness to carbonate hardness for the water, and the lime as sulphate to the lime as carbonate for the deposit. The significance of these ratios is discussed, and a classification of the different types of scale and sludge suggested. The chief types of scale are the gypsum and calcium carbonate scales, and mixtures of these, and the silica type. The sludges contain oxides of iron and aluminium, generally also silica and magnesia, and often organic matter. The various methods of preventing scale are examined, and the Cumberland electrolytic method more particularly discussed and recommended. S. I. LEVY.

Tentative standards [of specification and practice]. AMER. SOC. TESTING MATERIALS (Tentative standards, 1927, 824 pp.).—Tentative and revised specifications, tentative rules, methods of testing, and recommended practices are given for a number of commercial materials.

CHEMICAL ABSTRACTS.

[Standard specifications for] non-metallic materials. AMER. SOC. TESTING MATERIALS (Part II, Non-metallic materials, 1927, 1000 pp.).—Standard specifications, methods of testing, and definitions are given. CHEMICAL ABSTRACTS.

PATENTS.

Heat exchangers. SUPERHEATER Co., Assees. of W. H. ARMACOST (B.P. 286,682, 24.6.27. U.S., 9.3.27).—A number of standardised parts—tubular elements with circular ends in standard headers—are constructed so as to be suitable for varying conditions. The tubular elements in the intermediate part of their length are given a pseudo-oval section by branching them into two parallel circular tubes, capable of resisting considerable internal pressure. The elements may be rotated in the

headers so that the long axis of the cross-section is more or less athwart the stream of the other fluid.

B. M. VENABLES.

Heat-exchange apparatus. C. F. BRAUN (U.S.P. 1,662,143, 13.3.28. Appl., 28.3.23).—A series of horizontal tubes gives communication between chambers situated at the front and rear of a casing. These chambers are provided with partitions so that a fluid may flow backwards and forwards through the tubes between compartments in the front and rear chambers. A second fluid flows in at one end of the casing, passes around the tubes in the space between the front and rear chambers, and leaves at the opposite end. Means are interposed between groups of tubes for alternately increasing and then abruptly decreasing the velocity of the second fluid.

F. G. CLARKE.

Apparatus for the heating and cooling of fluids. C. W. STANCLIFFE (B.P. 286,757, 12.11.26 and 20.7.27).—In a heat exchanger mechanical means are provided to disturb the stationary film on the wall of the exchanger. *E.g.*, in a gear-wheel pump the gears themselves may be heated and idler gears provided to increase the squeezing action. In a tubular form of apparatus rotary brushes may wipe the surfaces of the tubes. B. M. VENABLES.

[Water-jacket for] shaft furnaces, gas producers, etc. STETTNER CHAMOTTE-FABR. A.-G., and E. TERRES (B.P. 284,639, 1.3.27. Ger., 2.2.27).—The water-jacket is formed at the lower part by a corrugated casing and at the upper part by an enlarged dome or head, and the three parts—combustion shaft, upper and lower jackets—are easily separable. B. M. VENABLES.

Grinding mills. L. MCG. FRASER (B.P. 287,318, 12.3.27).—A grinding pan or mill for use with volatile materials is provided with a vapour-tight cover vented only through a liquid seal or condenser; the latter device may be arranged to return condensed liquid into the mill. A closed cylinder and piston is used to withdraw the ground material and has two outlets—one for returning the material if not fine enough, the other for final discharge. B. M. VENABLES.

Centrifugal grinding mill. W. A. GIBSON and J. G. BURKE, Assrs. to BRADLEY PULVERIZER CO. (U.S.P. 1,663,881, 27.3.28. Appl., 22.6.25).—A mill having a horizontal annular grinding ring, co-operating centrifugally-actuated grinding members, and a superposed air-separating chamber has selectively operated, adjustable means for admitting air above and adjacent to the grinding ring, whereby the action of the air separator is regulated by admitting the air in predetermined directions relatively to the radius of the ring.

F. G. CLARKE.

Agitators or mixers. F. EPSTEIN (B.P. 279,127, 17.10.27. Belg., 16.10.26).—The apparatus is provided with loosely-slung baffles, counterweighted so that they can be caused to remain at the surface of the liquid or be submerged to any desired extent.

B. M. VENABLES.

Separation of subdivided materials. E. C. R. MARKS. From SUSQUEHANNA COLLIERIES CORP. (B.P. 286,951, 7.7.27).—The material is allowed to enter a circular chamber in which a fluid is flowing with vortical motion of sufficient violence to keep the stratified bed

of solids in rotary motion. The lighter material (and fluid) is drawn off through a central rising pipe, whilst the heavier passes out through the bottom to another separator if desired.

B. M. VENABLES.

Separation of intermixed divided materials of different specific gravities. R. PEALE, W. S. DAVIES, and W. S. WALLACE (B.P. 287,184, 13.9.26).—A shaking table is provided with a pervious bed through which air is blown upwards, to cause stratification. One of the strata—usually the heavier—is discharged by inclined guides over each long edge, and the other stratum proceeds straight on towards the end and is collected at a central opening.

B. M. VENABLES.

Drying of disintegrated material. O. SODERLUND, and TECHNO-CHEMICAL LABORATORIES, LTD. (B.P. 286,743, 7.10.26).—The air-borne material is passed up and down through a number of heated tubes or conduits, spreading and uniform distribution of the material being effected by changing the section of the conduits. Heating is preferably effected by a liquid and its vapour, *e.g.*, water and steam.

B. M. VENABLES.

Centrifugal apparatus for the treatment of sludge. E. C. ELSMORE, H. J. HOLFORD, and A. BISHOP (B.P. 286,855, 10.2. and 27.5.27).—The sludge is fed to the interior of a horizontal rotating drum lined with gauze or other filter medium; when a considerable amount of solid matter has accumulated, the rotation is stopped and a circular scraper with flexible edge is moved from end to end of the drum to discharge the solids through one end.

B. M. VENABLES.

Mixing of gases and liquids. E. PLAYER, and PARC ENGINEERING CO., LTD. (B.P. 286,812, 23.12.26 and 23.2.27).—The gas conduit is interrupted by a gap which is surrounded by a closed vessel containing the liquid. Dipping in the pool of liquid are one or more rotating toothed wheels which are spun by impingement of the current of gas and spray a quantity of the liquid into the gas stream.

B. M. VENABLES.

Apparatus for separating liquid containing a dispersed phase of another liquid. E. W. GARD, B. G. ALDRIDGE, and H. J. MULTER (U.S.P. 1,665,164, 3.4.28. Appl., 16.11.26).—One liquid dispersed in another is separated by passing the combined liquids through a succession of foraminous plates supported by grids which divide the separating chamber into sections. The combined liquids enter at one end of the chamber, and the liquid which passes more readily through the plates passes through all the latter and leaves through an outlet at the opposite end, whilst the other liquid leaves the chamber by one or other of the outlets placed between the grids.

F. G. CLARKE.

Centrifugal thickening of mixtures and clarifying of liquids. H. M. CHANCE (U.S.P. 1,664,769, 3.4.28. Appl., 29.7.25).—The fluid mixture of liquid and comminuted solids passes through the perforated walls of a drum rotating at high speed. The solids proceed into a region of relatively slow rotation between the drum and a casing, whilst the liquid moves towards the centre of rotation. Clarified or partly clarified liquid may thus be withdrawn from the drum and the

thickened mixture from the region between the drum and the casing. F. G. CLARKE.

Preparation of filtering materials. F. H. WEIL, Assr. to SEITZ-WERKE G.M.B.H. (U.S.P. 1,664,275, 27.3.28. Appl., 24.2.27. Ger., 22.5.24).—Natural asbestos is treated under pressure with a non-reducing gas which unites with the impurities in the asbestos forming compounds having no action on the liquids to be filtered. F. G. CLARKE.

Evaporating apparatus. AKTIEBOLAGET SVENSKA MASKINVERKEN, and E. G. ERIKSSON (B.P. 286,982, 6.9.27).—The liquid to be evaporated flows from end to end in the lower part of a fixed horizontal drum, within which is an inner, rotating, heating drum provided with disc-like projections. Within the discs the heating medium (steam) is caused to flow in spiral passages, or the discs may be formed as spiral steam coils. The steam is supplied to the larger part of the inner drum, which is provided with individual inlets to each spiral coil, and the outlets for condensate from the coils communicate with an outlet conduit co-axial with the steam drum. At the bottom of the fixed drum are screens upstanding between the heating discs and a conveyor for slime.

B. M. VENABLES.

Bag filters. MASCHINENFABR. BETH A.-G. (B.P. 281,994, 27.6.27. Ger., 11.12.26).—The filter bags are divided into groups, and two fans are mounted on a common shaft, one for drawing the main stream of gas through the filter, the other for forcing reverse-current cleaning air through one group only (at a time) of the filter. The dust dislodged from this group falls into a conveyor and the air joins the main stream and therefore passes through the other groups in the filtering direction. B. M. VENABLES.

Purification of furnace gases. C. V. A. ELEY (B.P. 285,544, 15.11.26).—The discharge of smoke or noxious gases from furnaces is reduced or eliminated by passing the gases successively through a filter chamber and a tank. The filter chamber may be provided with trays containing chemical reagents for purifying the gases, or may contain a spray for supplying water or other liquid for the same purpose. The gases enter the tank below the level of the liquid and may be made to pass below a baffle plate attached to the inlet pipe before passing up through the liquid and escaping. The tank is provided with a sump containing a helical conveyor for the removal of deposited solid matter, which passes through a valve into a chamber from which it can be removed from time to time. A. B. MANNING.

Gas-cleaning apparatus. C. G. HAWLEY, Assr. to CENTRIFIX CORP. (U.S.P. 1,663,597, 27.3.28. Appl., 4.5.25).—The gas passes from a surrounding casing into an annular tuyère consisting of vertical blades set tangentially between an upper and a lower ring member so that the overlap is adjustable. Communicating with the tuyère through the top of the casing is a conduit for the gas, and immediately below the tuyère is a central disc which deflects the dust into a receiver placed below.

F. G. CLARKE.

Extraction of extraneous material from fluids or gases. J. GORDON & Co., LTD. From HAGAN CORP. (B.P. 286,853, 9.2.27).—A form of apparatus

without moving parts suitable for separating dry steam from wet steam is described. B. M. VENABLES.

Fire-extinguishing composition. AMDYCO CORP., Asses. of F. L. DUNLAP and N. T. EWER (B.P. 269,879, 1.4.27. U.S., 21.4.26).—A composition which reacts in the presence of water to produce a stable foam comprises aluminium sulphate containing not more than 14 mols. of water of crystallisation, sodium bicarbonate, a stabiliser, and an inert filler, the ingredients being ground to pass 40-mesh. The proportion of water of crystallisation in the aluminium sulphate may be reduced to 7 mols., and the weight of the inert material increased proportionately. L. A. COLES.

Manufacture of ice crystals. S. C. CARNEY, Assr. to ROXANA PETROLEUM CORP. (U.S.P. 1,664,850, 3.4.28. Appl., 4.2.26).—Separated quantities of ice are passed through gaseous carbon dioxide under pressure and then moulded by being pressed together while impregnated with the gas. H. ROYAL-DAWSON.

Stabilisation of chemical substances. H. G. GRIMM (U.S.P. 1,664,678, 3.4.28. Appl., 29.4.24. Ger., 5.5.23).—See B.P. 240,884; B., 1925, 977.

Heat interchangers. A. E. LEEK (B.P. 270,250, 10.1.27. Ger., 1.5.26).

Separation of granular materials (B.P. 287,262).—See II. **Sublimation** (U.S.P. 1,662,056).—See III. **Drying ammonium salts etc.** (B.P. 277,652).—See VII. **Tunnel kiln** (U.S.P. 1,662,063).—See VIII. **Filling cracks in brickwork** (B.P. 286,933).—See IX. **Mixing machine** (B.P. 287,016).—See X.

II.—FUEL; GAS; TAR; MINERAL OILS.

Pressure extraction of coal with tetralin. E. BERL and H. SCHILDWÄCHTER (Brennstoff-Chem., 1928, 9, 105—113).—Two bituminous coals have been extracted with tetralin under pressure at 250° and the extracts examined. Carbonisation of the residue yielded only a small quantity of tar, showing that the constituents of the coal soluble in tetralin were the main source of the tar obtained on carbonisation. One extract, representing 20.3% of a "flaming gas coal," was treated with ether, which dissolved 54.9%, and the soluble fraction was separated into acidic and basic constituents (1.55% and 0.023%, respectively, of the total extract), asphalts precipitated by light petroleum (8.87%), and neutral oils (31.1%); the remainder of the soluble fraction consisted of solids precipitated by the acid and alkali used for extraction. Separation of the neutral oils by the Edeleanu process showed them to consist of about 20% of saturated and 80% of unsaturated hydrocarbons. From the saturated fraction solid paraffins of composition $C_{22}H_{46}$, $C_{23}H_{50}$, and $C_{30}H_{62}$ (or possibly $C_{30}H_{60}$) were separated in crystalline form. The unsaturated fraction contained hydrocarbons of the series C_nH_{2n-4} to C_nH_{2n-18} , the value of n ranging from 12 to 26. The asphalts precipitated by light petroleum were probably of aromatic nature; they could be nitrated, were oxidised to acids by alkaline permanganate, and gave Nastjukov's reaction. The extract obtained from the other coal amounted to 17% and behaved very similarly on examination, although a slightly different procedure

was adopted, the ether extraction being preceded by a separation into saturated and unsaturated constituents by treatment with liquid sulphur dioxide.

A. B. MANNING.

Dry distillation of lignin obtained from beech, oak, and birch. G. SZÉLENYI and A. GÖMÖRY (*Brennstoff-Chem.*, 1928, 9, 73—77).—Lignin prepared from these woods by Kürschner's process (cf. B., 1925, 912), and purified by a second hydrolysis of 24 hrs., has a methoxy-content (Zeisel) of 14.5—14.86%, 14.94—15.65%, and 14.20—14.65% for beech, oak, and birch, respectively, the yield of lignin in each case amounting to 18—19%. On dry distillation the three lignins afford qualitatively the same products as the distillation of lignin from aspen or pine wood (cf. Heuser and Skiöldebrand, B., 1919, 215 A; Heuser and Brötz, B., 1925, 585). The yield of acetic acid is practically the same in all cases, but the yield of methyl alcohol is about 66% higher with oak- and beech-lignin and 14% higher with birch-lignin than with lignin from the pine or ash. The yields of tar are lower, beech-lignin giving only 30%, oak 23%, and birch-lignin only 40% of that obtained from pine-wood lignin. Lignins from pine- and beech-wood give practically the same yield of acetone, slightly higher yields being obtained in the case of lignin derived from the oak, beech, or aspen. Only 10.15% of the original methoxy-content of the lignin is found in the liquid products in the case of beech- and oak-lignin, and only 7.9% in the case of birch-lignin. The yield of gaseous products was approximately the same in all three cases. Oak- and beech-lignin afford 53% of coke, birch-lignin 51%, these yields being about 15% higher than that obtained from pine-lignin.

R. BRIGHTMAN.

Carbonisation experiments at the Jena gas-works. GÜLICH (*Gas- u. Wasserfach*, 1928, 71, 8—10).—The formation of a layer difficultly permeable to gas at the top of the column of coal in a vertical retort, which leads to loss due to increased gas pressure in the lower part of the retort, is avoided by the use of a layer of coke on the top of the coal. Experiments on the maintenance of a gas of constant calorific value by control of the steaming and the amount of suction are briefly described.

A. B. MANNING.

Simultaneous removal of ammonia and sulphur compounds from carbonisation gas. H. BÄHR (*Gas- u. Wasserfach*, 1928, 71, 169—173, 201—210).—The recovery, from low-temperature carbonisation or coke-oven gases, of ammonia and sulphur compounds by the Feld and the Burkheiser processes is discussed, and a new process, which has been subjected to laboratory and semi-technical tests, is described. The gas from the retorts is first cooled to 20—25°, and then subjected to electrical de-tarring. The tar is separated from the condensate, which then contains most of the ammonia and hydrogen sulphide. The liquor is distilled, and the ammonia and hydrogen sulphide evolved are passed back into the de-tarred gas. About 5—6% of air is admitted, and the mixture passed over a catalyst (unspecified), whereby the hydrogen sulphide is oxidised to sulphur dioxide. By a proper arrangement of heat exchangers the oxidation reaction may be made

autothermic, the gases requiring to be heated before commencing the operation. The resulting gas is cooled, when most of the sulphur dioxide reacts with ammonia and is recovered as ammonium sulphite, the last traces of which are removed by electrical precipitation. Any excess ammonia is recovered from coolers as ammonia liquor, and is of sufficient purity to be used in an ammonia oxidation plant. Alternative methods for treating the ammonium sulphite are discussed, but in general it is proposed to volatilise it with air and then to pass the mixture through an ammonia oxidation converter, the resulting oxides of nitrogen and sulphur being removed in condensers as nitrosylsulphuric acid (subsequently converted into mixed nitric and sulphuric acids) or passed for recovery to a form of lead chamber sulphuric acid plant. A flow-sheet with estimates of capital and manufacturing costs for a plant to treat the gas from the distillation of 400 tons of coal per day is given, and it is claimed that the new method of treatment is 25—50% cheaper than the older ones.

A. E. MITCHELL.

Dry purification [of gas]. G. OFFE (*Gas- u. Wasserfach*, 1928, 71, 222—224).—When fresh bog-ore is used for purification of gas the material often forms large lumps and eventually sets up considerable back-pressure. This agglomeration is due to the high activity of some parts of the oxide, and in these places the temperature will rise and the evaporation of water will be more pronounced; the same effect is produced owing to the impossibility of filling the boxes so that the gas passes evenly throughout the purifying mass. Fresh oxide is not so useful in removing cyanogen from the gas as some which has been partly fouled; this is due to its low content of ferrous iron, which is necessary in the formation of Berlin-blue. A mixture of 3 pts. of fresh bog-ore with 1 pt. of oxide from boxes which have been in use has been found satisfactory for the removal of hydrogen sulphide and cyanogen compounds.

R. H. GRIFFITH.

Reduction of carbon monoxide and dioxide with hydrogen in the hot-cold tube at ordinary and high pressure. F. FISCHER and (Führn.) VON WANGENHEIM (*Brennstoff-Chem.*, 1928, 9, 94—97).—Results previously obtained by Fischer and Jaeger (B., 1926, 777) have been confirmed with various binary and ternary mixtures of carbon monoxide, carbon dioxide, and hydrogen. Reduction is perceptible at 500°, and is accelerated by higher temperatures and pressures. Covering the iron spiral with the asbestos or with aluminium hydroxide had little effect. Methane is formed in amounts up to 30%, and is accompanied by higher homologues. Formaldehyde was only detected qualitatively, and methyl alcohol was not formed. Appreciable amounts of carbon were deposited. On replacement of the iron spiral by a copper spiral or foil, partial reduction of carbon dioxide to carbon monoxide was observed only at 800°; at lower temperatures no reaction occurred. Nickel wire gives similar results to iron, but the reaction is slower. Tungsten, molybdenum, and charcoal behave similarly above 550°, but the reaction velocity is considerably less than in the case of iron and nickel.

R. BRIGHTMAN.

Gas reactions in the hot-cold tube. F. FISCHER and (Frhrn.) VON WANGENHEIM (Brennstoff-Chem., 1928, 9, 97—98).—When heated at 600—700° and under 1—6 atm. in the hot-cold tube previously described (B., 1926, 777) in presence of steam and iron, carbon monoxide is decomposed into carbon dioxide and carbon and into carbon dioxide and hydrogen, the former reaction preponderating. The same reactions occur in the presence of methane, the latter gas being practically unchanged. Mixtures of carbon dioxide and methane are practically unchanged under these conditions. Methane alone is practically unchanged, only traces of carbon being deposited and a very little hydrogen formed, although condensation to form higher homologues may occur. Unsaturated hydrocarbons, formaldehyde, or alcohols were not formed.

R. BRIGHTMAN.

Testing laboratory of the American Gas Association. R. M. CONNER and F. E. VANDAVEER (Ind. Eng. Chem., 1928, 20, 307—310).—Small amounts of carbon monoxide are determined by the iodine pentoxide method, by thermal conductivity measurement, and by a recording apparatus depending on the liberation of heat in the selective oxidation of carbon monoxide by a mixture of copper oxide and manganese dioxide at 100°. All these methods are capable of an accuracy of 0.002%. The maximum proportions of carbon monoxide allowable in the gases of combustion of different types of gas apparatus are discussed.

C. IRWIN.

Variation in consistency of tars with temperature.

(A) H. M. SPIERS (Brennstoff-Chem., 1928, 9, 77—78). (B) H. MALLISON and F. SOLTAN (ibid., 78—79). (C) H. M. SPIERS (ibid., 79).—(A) The formula $\log(C_1/C_2) = K(T_2 - T_1)$, expressing the relation between temperature and consistency of tars (cf. Mallison and Soltan, B., 1927, 517) is accurate only within the limits of experimental error and within a temperature range of 10°. The value of K changes suddenly at a definite temperature, depending on the nature of the tar, and this temperature corresponds with a pronounced change in the physical properties of the tar.

(B) Spiers' remarks (preceding) are criticised. On account of the limited applicability of Spiers' formula, four or six consistency determinations may be necessary for a particular tar to establish values for K , and the authors suggest making a direct determination of consistency at standard temperature (25°). The factor K , being a completely atypical quantity, is insufficient for characterisation of the consistency curve, and the mathematical treatment of the consistency curve of tars is regarded as without practical interest.

(C) A reply. In the great majority of cases the consistency curve shows no break between 24° and 34°, and this temperature range in which K remains constant is sufficiently wide to allow of calculations of consistency at 25° with reasonable accuracy. The calculation of consistency from measurements at two arbitrarily chosen temperatures affords a valuable check on the direct determination. The method offers the further advantage that technical controls can be carried out on the hot tar at temperatures above 25°. In factory work one species of tar only is usually dealt with, and, the properties of this tar being once established,

working temperatures can be chosen between which the consistency curve shows no break. The anomalous value of 55 sec. at 27°, referred to by Mallison and Soltan, disappears if the observation is made at 26° and not at 27°, the observed and calculated consistency values being then 55 and 56.6 sec., respectively.

R. BRIGHTMAN.

Utilisation of *o*-cresol as a necessary condition for the working-up of medium tar oils. F. BOVINI (Notiz. chim.-ind., 1927, 2, 688—690).—A search for new uses for *o*-cresol. Dyes were prepared from *o*-cresol (G.P. 101,541, 102,897) and from *p*-nitroso-*o*-cresol (G.P. 197,165, 205,882), and the results of variations in the methods of preparation are recorded. *p*-Nitroso-phenol is best prepared by adding phenol (94 g.), 25% aqueous sodium hydroxide (80 c.c.), sodium nitrite (75 g.), and water (300 c.c.), during 2 hrs. to 93% sulphuric acid (90 g.), water (300 c.c.), and ice (400 g.), the temperature not exceeding 4°; during the addition 10% aqueous sodium hydrogen sulphite (100 c.c.) is slowly added. The mixture is agitated for at least 1 hr., filtered rapidly at 10°, washed with ice-water (100 c.c.), and dried in air at 40—50°. The yield is 75%. *p*-Nitroso-*o*-cresol was similarly prepared in 80—85% yield.

CHEMICAL ABSTRACTS.

Purification of effluent liquors from brown coal low-temperature distillation plants. D. WITT and F. SCHUSTER (Gas- u. Wasserfach, 1928, 71, 241—244).—It is pointed out that although these effluents contain ammonia, pyrocatechol, acetic acid, and phenols, together with hydrogen sulphide, the quantities involved are so small as to render unwarrantable any large capital or operating expenditure for their purification. Various proposed methods of purification are discussed, and dismissed owing to their high costs, and an account of experiments made at the Berlin gas-works is given. The electrolytic process was abandoned in favour of one in which the alkaline effluent liquor is first filtered through a bed of the coke obtained in the process to remove hydrogen sulphide. The filtrate is then acidified by treatment with the gas containing sulphur dioxide from the brown coal generators. A second filtration through brown coal coke then purifies the effluent sufficiently for it to be allowed to pass out.

A. E. MITCHELL.

Asphaltogenic substances in American lubricating oils. L. A. BASS and F. C. VILBRANDT (J. Elisha Mitchell Sci. Soc., 1927, 43, 21).—The European asphalt test indicates that American oils do not contain asphaltogenic substances in appreciable quantities; the test may be invalid.

CHEMICAL ABSTRACTS.

Solubility of paraffin wax in pure hydrocarbons. P. WEBER and H. L. DUNLAP (Ind. Eng. Chem., 1928, 20, 383—384).—The solubility of paraffin wax in pentane, hexane, heptane, octane, and isodecane was determined at temperatures from 0° to 25°, and was found to increase from isodecane to pentane; the increase with rise of temperature was more rapid with the solvent of higher mol. wt. It is probable from the results, expressed as g. of paraffin per mol. of solvent, that the mol. ratios of solvents to solute may be constant, and that a definite number of molecules of solvent are associated with 1 mol. of solute.

D. G. HEWER.

Heat transfer in pipes. MORRIS and WHITMAN. **Counter-current absorption systems.** LEWIS and McADAMS.—See I. **Decay of oakwood.** BRANDL.—See V. **Wood preservatives.** DEHNST.—See IX. **Utilisation of acid sludge.** LIKHUSHIN.—See XII. **Nitrogen of peats and humus soils.** ELLIS and MORISON. **Insecticides.** FRANÇOIS and SEGUIN.—See XVI.

PATENTS.

Coke ovens. R. E. ELLIS. From FOUNDATION OVEN CORP. (B.P. 286,776, 8.12.26. Addn. to B.P. 279,955; B., 1928, 114).—The modification consists in admitting air to the regenerators from flues disposed at the bottom thereof, and passing the air vertically upward through the regenerators to the combustion flues.

A. B. MANNING.

Separation of powdered or granular material, especially coal. R. LESSING (B.P. 287,262, 11.1.27).—A current of gas is caused to flow up a tubular vessel into which the coal is fed and down which the coarse coal drops against the gas current. Later on an increase of cross-section takes place, preferably at an inclined portion of the tube, and middlings drop out; the dust is finally separated and the gas re-used. The middlings may be retreated in a separate unit. B. M. VENABLES.

Treatment of materials with binders and briquetting said material. R. LESSING (B.P. 286,336, 3.11.26).—Finely-divided coal, coke, etc. is thoroughly mixed with dehydrated coal tar, and the mixture extracted with petroleum spirit in such a way that the pitch is precipitated on the solid particles of the material, which is then freed from any remaining solvent by steam-distillation and conveyed directly to the briquetting machine. Addition of a small proportion of sulphuric acid to the mixture before extraction facilitates the process. The petroleum spirit is recovered by distillation from the solution containing tar oils, and is used in the treatment of a further quantity of the material. The briquettes are improved by finally submitting them to a baking process.

A. B. MANNING.

Distillation of coal and similar fuels. H. M. RIDGE (B.P. 286,104, 23.8.26).—Coal, previously reduced to a suitable degree of fineness, traverses a succession of superposed horizontal hearths in an evenly distributed layer. Rabbling devices stir the coal continuously and feed it from each hearth to the one immediately below. The hearths are heated by gas-fired ducts underneath them. The coked residue finally traverses a cooling hearth, the walls of which are provided with air ducts, and is briquetted immediately on its discharge from the retort. The volatile distillation products from each hearth are separately condensed. Those from the lowermost hearths may be returned to the coke in the cooling hearth in order to facilitate the briquetting of the residue.

A. B. MANNING.

Distillation of carbonaceous or bituminous substances. W. E. EVANS. From KOHLENVEREDLUNG A.-G. (B.P. 286,404, 31.12.26).—The carbonaceous material is fed in the form of a powder to a nozzle of suitable form through which is blown hot generator- or water-gas directly from the producer. The intimate

mixture of gas and powdered material so produced is carried by the gas current through a chamber in which the material undergoes distillation by the sensible heat of the gas. The solid residue is separated from the volatile products of distillation and used as fuel in the gas producer. In one form of apparatus the retorts have vertical shafts, and can be arranged so that the material traverses two or more of them in succession; the retorts in another form have a number of parallel, slightly inclined or horizontal tubes, which may be heated externally if desired.

A. B. MANNING.

Distillation of solid carbonaceous materials. H. NIELSEN and B. LAING (B.P. 287,037, 4.12.26 and 21.2.27).—Solid carbonaceous materials are distilled in a current of an inert, gaseous, heating medium, the volume of which is so great that the heaviest oil fractions produced in the distillation are completely vaporised at temperatures not exceeding 450–550°. With materials such as oil shales containing a large percentage of oil, the volume of distilling medium used is such that it may be cooled to 80–100° without causing condensation of the heaviest fractions. Coking coals are subjected to a preliminary treatment by gases containing an oxidising constituent in order to reduce their caking properties. By the addition of some oxygen to the distilling medium the resin-forming constituents of the oils are polymerised and the subsequent refining processes thereby facilitated. The higher-boiling fractions of the refined neutral oils yield paraffin wax and valuable lubricating oils. The solid residue forms a free-burning fuel containing 4–15% of volatile matter.

A. B. MANNING.

Production of high-value oils from raw coal by low-temperature distillation and hydrogenation. R. FEIGE (B.P. 274,465, 11.7.27. Ger., 16.7.26).—In a process for the economical production of oils from coal, the coke produced is used in part for the generation of electric current, which is in turn used to electrolyse water. The remaining coke is gasified with the electrolytic oxygen, the hydrogen being used to hydrogenate the resulting products, which may or may not be previously mixed with gas and/or tar from the coal-distillation plant.

C. O. HARVEY.

Destructive hydrogenation of carbonaceous materials. I. G. FARBERIND. A.-G. (B.P. 272,538, 10.6.27. Ger., 11.6.26).—The gases from processes for the hydrogenation of carbonaceous materials at high temperatures and pressures are scrubbed, under pressure, with benzine, preferably that obtained in the process. The gaseous hydrocarbons are thereby scrubbed out and the residual gases can be recirculated.

A. B. MANNING.

Production of [unsaturated] hydrocarbons from coal, tars, mineral oils, etc. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 286,825, 3.1.27).—Valuable unsaturated hydrocarbons, in particular acetylene, are produced by submitting coal, tars, oils, etc., either vaporised or in a finely-divided condition, to the action of an electric arc in the presence of less than half the quantity of water vapour requisite for the production of water-gas. The water vapour may be replaced by

hydrogen, nitrogen, carbon dioxide, etc., and the mixture fed to the arc is preferably preheated.

A. B. MANNING.

Production of carbon. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 286,845, 4.2.27).—A continuous process for the manufacture of carbon from carbon monoxide consists in adding 0.1—0.2% (by vol.) of iron carbonyl vapour to the gas and passing the mixture through a high-pressure furnace at about 400°. The effluent gas can first be freed from carbon dioxide by washing with water under pressure, and then subjected to the same process in a second apparatus. The carbon formed contains about 2% Fe.

A. B. MANNING.

Fuels for internal-combustion engines. BRIT. DYESTUFFS CORP., LTD., and S. COFFEY (B.P. 287,192, 25.10.26).—Heavy-metal derivatives of β -diketones of the type $R \cdot CO \cdot CHR' \cdot CO \cdot R''$, where R and R'' are hydrocarbons or substituted hydrocarbon radicals, and R' is the same or hydrogen, are added to motor fuels as antidetonants. Chromium, iron, cobalt, manganese, and thorium derivatives of acetylacetone, propionylacetone, and γ -methylacetylacetone are particularly claimed.

C. HOLLINS.

Manufacture of pulverulent fuel for internal-combustion engines. I. G. FARBERIND. A.-G. (B.P. 270,702, 2.5.27. Ger., 7.5.26).—Pulverulent fuels are charged with gaseous, or vaporised, or atomised liquid fuels. Their ignition temperature is thereby reduced and their speed of combustion raised.

A. B. MANNING.

Motor fuel. T. MIDGLEY, JUN., ASSR. to GEN. MOTORS CORP. (U.S.P. 1,662,323, 13.3.28. Appl., 16.5.25).—The fuel is comprised of a low-compression fuel and nickel carbonyl.

F. G. CLARKE.

Gas producer for use with wood etc. G. IMBERT (B.P. 279,475, 20.10.27. Ger., 25.10.26).—A producer suitable for use on motor vehicles.

A. B. MANNING.

Regenerator settings for use in the manufacture of gas. R. W. BROADHEAD (B.P. 286,418, 17.1.27).—The producer and regenerator are arranged externally to the retort bench, one regenerator serving a number of beds of retorts, the waste gases from which pass by insulated ducts to the regenerator, whilst preheated producer gas and secondary air are led back by other insulated ducts to the beds of retorts.

A. B. MANNING.

Production of rich gas and semi-coke from bituminous fuel. A. L. MOND. From METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 285,664, 15.3.27).—The fuel is passed continuously down a column in the upper zone of which it is distilled by the sensible heat of the gas produced by the gasification in the lower zone of part of the semi-coke. The gasification process is carried out by means of oxygen, or air rich in oxygen, and steam if desired, in such a manner as to produce only the sensible heat required for the distillation in the upper zone; the remainder of the semi-coke is withdrawn without taking part in the gasification process. The gas may be enriched by injecting tar residues etc. into the hot zones of the generator.

A. B. MANNING.

Production of coal gas. W. M. CARR (B.P. 286,758, 13.11.26).—The proportion of a diluent gas added to coal gas to maintain a constant calorific value is regulated by an electrical or other device controlled by the flame temperature of a burner supplied from a by-pass through which a continuous rapid flow of the diluted gas is maintained.

A. B. MANNING.

Gas-purification process and apparatus. KOPPERS Co., and F. W. SPERR, JUN. (B.P. 261,755, 17.11.26. U.S., 18.11.25. Cf. B.P. 255,139—255,145; B., 1926, 813).—Hydrogen sulphide is removed from coke-oven or other gases by a cyclic process in which the gases are treated with wash liquors containing ammonia and a suspension of hydrated ferric oxide or similar metallic compound. If the gases contain ammonia this may be utilised in the preparation of the ferric oxide suspension from a solution of an iron compound. After absorption of the hydrogen sulphide the ammonia is recovered from the sulphided liquor by distillation; the liquor is then aerated, the ferric sulphide being converted into ferric oxide and sulphur, and, after separation of the sulphur, is recirculated, if necessary with part or all of the recovered ammonia, for further absorption of hydrogen sulphide. The process may be used in conjunction with either the "indirect" or "direct" system of ammonia recovery. In the former case the whole of the ammonia is absorbed in the wash liquor; in the latter, the temperature of the liquor is adjusted so that a part only of the ammonia is absorbed, the remainder being removed subsequently in a saturator containing sulphuric acid. Gases containing little or no ammonia are scrubbed in a tower at the top of which is introduced the liquor containing the iron compound, and at part of the way up which is introduced an ammoniacal solution. From time to time part of the liquor may be withdrawn from circulation after the aeration and oxidation stage, and treated for the recovery of ammonia present as fixed salts. Details are given of complete plants suitable for the operation of the processes.

A. B. MANNING.

Absorption of fluids from gases. Removal of tar from gases. E. PIRON, ASSR. to PIRON COAL DISTILLATION SYSTEMS, INC. (U.S.P. 1,664,483—4, 3.4.28. Appl., [A] 30.9.21, [B] 25.3.25).—(A) An absorption device consists of one or more units each comprising a chamber containing a horizontal, foraminous member across which a mentrum flows in separate streams through guide-ways from an inlet at one side to an outlet at the other. (B) Wood-distillation gases are scrubbed with a medium inert towards tar, whereby the greater part of the latter is removed. After heating the partially cleansed gases by mixing them with hot residual gases, they are scrubbed with sulphuric acid at such a temperature that no constituent other than tar condenses. Pyroligneous acid is then condensed and a sufficient quantity of the residual gases is heated to give the necessary temperature during the acid washing.

F. G. CLARKE.

[Tar] still. F. PUENING, ASSR. to KOPPERS Co. (U.S.P. 1,664,863, 3.4.28. Appl., 25.7.24).—In a vertical cylindrical still for continuous use, baffling effects are brought about by means of a diametrically placed

partition and by baffle plates, including an inner plate concentric with the still wall. C. O. HARVEY.

Separation of acid sludge emulsions. Hydrolysis of acid sludge. E. W. ROTH, Assr. to GEN. PETROLEUM CORP. OF CALIFORNIA (U.S.P. 1,665,189 and 1,665,190, 3.4.28. Appl. [A, B], 13.4.27).—(A) Emulsions of tar and dilute acid resulting from the hydrolysis of acid sludge are broken by passing through a pack of granular inert material followed by settling and separating operations. (B) Acid sludge is separated into useful constituents by digestion with a solvent oil and dilute sulphuric acid of a density initially greater than that of the sludge. C. O. HARVEY.

Fire still and oil treater. F. E. GILMORE, Assr. to F. E. GILMORE Co. (U.S.P. 1,664,920, 3.4.28. Appl., 4.2.26).—The oil is evaporated from a chamber heated by a central cylindrical flue fitted with radiating fins and having a burner at its base. C. O. HARVEY.

[Oil] evaporator. C. F. BRAUN (U.S.P. 1,662,142, 13.3.28. Appl., 14.3.22).—A casing contains a series of vertical tubes upon the surfaces of which flow films of the oil to be distilled. Steam passes up around the tubes, and oil, always at a higher temperature than the steam, is passed down through the tubes. F. G. CLARKE.

Cracking of hydrocarbon oils or their distillates by distillation under pressure. S. STRANSKY and F. HANSGIRG (B.P. 267,958, 17.3.27. Austr., 18.3.26).—In cracking hydrocarbon oils, the formation of gaseous and coke-like products is minimised by maintaining the oil at a temperature just sufficient for the generation of the necessary pressure (about 400°). Under these conditions, splitting of the paraffins and naphthenes of high mol. wt. proceeds to an equilibrium point, the reactions involved being reversible. In order to urge the cracking process towards completion, the cracked products are rapidly removed from the reaction zone by means of electrical resistance heaters situated inside the still and capable of providing a great quantity of heat per unit of time. The level of the oil in the still is also automatically adjusted, as, with an overfilled still, the vapours carry over with them entrained droplets of the heavier hydrocarbons, and, with a low level of oil, the necessary pressure cannot be attained without applying sufficient heat to vaporise the higher-boiling constituents. C. O. HARVEY.

Cracking of hydrocarbon oil. R. T. POLLOCK (B.P. 287,344, 29.4.27).—In a cracking plant consisting of a tubular still connected with an expansion chamber, a dephlegmator, and condensing apparatus, the reflux from the dephlegmator, prior to its return to the cracking still, is freed from more volatile constituents in a small still heated by the flue gases from the main still. C. O. HARVEY.

Cracking of hydrocarbons. G. H. TABER, JUN., Assr. to SINCLAIR REFINING Co. (U.S.P. 1,663,868, 27.3.28. Appl., 24.6.26).—The uncondensed vapours remaining after the passage of the products of a cracking operation through a condenser are partially retained in an absorbent medium and subsequently recovered. Unabsorbed vapours and gases are finally treated with some of the cracking stock. C. O. HARVEY.

Treatment of heavy hydrocarbons for the production of lighter hydrocarbons. C. ARNOLD. From STANDARD DEVELOPMENT Co. (B.P. 286,917, 2.5.27).—In a cracking system involving rapid passage of the oil through heating chambers, wherein the necessary temperature is attained with a minimum of actual decomposition or carbon formation, to a series of conversion chambers and thence to the vaporisation chambers, individual conversion chambers may be isolated for cleaning purposes without interfering with the continuity of the process. A lower cracking temperature than usual may be employed, as the time of passage of the oil through the series of converters is prolonged. The cleaned converters may be charged with raw material before being reintroduced into the system. C. O. HARVEY.

Cracking of oil. E. C. HERTHEL, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,660,647, 28.2.28. Appl., 23.3.25. Renewed 26.4.27).—The furnace gases employed to heat a pressure still are first led around an auxiliary coil, through which tar-free oil from the reflux is returned to the still, and are then further cooled by dilution with a portion of the waste furnace gases. Overheating of the still with consequent deposition of carbon is avoided. T. S. WHEELER.

Cracking of oils. F. PUENING (U.S.P. 1,664,263, 27.3.28. Appl., 26.5.19).—The oil is heated and cracked while circulating through the annular space between two concentric drums provided with scrapers for the continuous removal of deposited carbon. C. O. HARVEY.

Distillation of oils occurring in the petroleum, tar, and similar industries, more particularly for the production of lubricants. L. STEINSCHNEIDER (B.P. 264,476, 23.12.26. Czechoslov., 15.1.26).—An apparatus for removing from distilled lubricating oils the low-boiling and dissolved gaseous decomposition products consists of two interconnected evaporators from the first of which the volatile impurities are withdrawn. The pressure in the second evaporator, from which the purified oil is distilled, is lower than in the first. The heating of the oil to the final temperature may be carried out in stages by employing a battery of these distilling units. C. O. HARVEY.

Distillation of oil. H. L. DOHERTY, Assr. to HEAT TREATING Co. (U.S.P. 1,662,105, 13.3.28. Appl., 7.7.20).—Fractions of progressively rising b.p. are obtained by passing the oil in a downward direction through a chambered still. C. O. HARVEY.

Distillation of lubricating oils. R. W. HANNA, Assr. to STANDARD OIL Co. OF CALIFORNIA (U.S.P. 1,664,977, 3.4.28. Appl., 4.10.23).—The oil is heated to a cracking temperature and maintained in the liquid state. Before substantial cracking occurs the heated oil rapidly passes to a vaporising chamber maintained under reduced pressure, wherein the temperature of the oil drops below the cracking point. The vapours, thus freed from "mist" without substantial scrubbing, are fractionally condensed. C. O. HARVEY.

Neutralising treatment of acid oils for lubricant stocks. Treatment of lubricant stocks. G. F. OLSEN, Assr. to GEN. PETROLEUM CORP. OF CALIFORNIA

(U.S.P. 1,665,110—1, 3.4.28. Appl., [A] 31.8.26, [B] 15.10.27).—(A) Oils containing organic and mineral acid are agitated and treated with sufficient pulverised solid alkali to neutralise only the mineral acid, the addition of water being avoided throughout. (B) In treating viscous petroleum oils with liquid sulphur dioxide, the oil is previously diluted with lighter petroleum products.

C. O. HARVEY.

Manufacture from bituminous shales of a product adapted for dry distillation. PATENTAKTIEBOLAGET GRÖNDAL-RAMÉN (B.P. 278,378, 30.9.27. Swed., 2.10.26).—Shales high in bitumen are finely crushed, thoroughly mixed to give a homogeneous product, and the latter is formed into balls by further admixture with water and passage of the moist material through a revolving drum. The balls are dried and strengthened by treatment with hot fuel gases in a second drum.

A. B. MANNING.

Purification of the liquid complex hydrocarbon products of the destructive hydrogenation of carbonaceous materials. I. G. FARBERIND. A.-G. (B.P. 257,270, 18.8.26. Ger., 20.8.25).—The crude liquid hydrogenation products are treated with one of the lower aliphatic alcohols (e.g., crude synthetic methyl alcohol obtained by the catalytic hydrogenation of carbon monoxide). The treatment is preferably carried out by a counter-current method, the solid paraffins having first been separated from the crude oils at -10° . The impurities dissolve in the alcohol and highly-refined products are obtained, suitable for use as lubricating oils etc.

A. B. MANNING.

Recovery of paraffin [from slack wax]. W. A. GRUSE and W. F. FARAGHER, ASSRS. to GULF REFINING Co. (U.S.P. 1,663,592, 27.3.28. Appl., 28.10.24).—Hard paraffin wax is recovered from solidified slack wax by passing the latter through a mass of water maintained at a suitable temperature and repeatedly cutting the wax so as to enable the oil to separate therefrom and rise to the surface.

C. O. HARVEY.

Disposal of spent clay in [oil]-refining plants. J. M. CORY and F. H. BUNKE, ASSRS. to SOLAR REFINING Co. (U.S.P. 1,660,434, 28.2.28. Appl., 27.1.26).—A mixture of the clay and a hydrocarbon oil is claimed as a liquid fuel.

T. S. WHEELER.

Washing apparatus for coals. P. WOLF (U.S.P. 1,666,189, 17.4.28. Appl., 28.6.26. Fr., 3.7.25).—See B.P. 254,709; B., 1927, 161.

Degasification of coal. N. YOUNG (U.S.P. 1,664,723, 3.4.28. Appl., 30.10.19. Ger., 1.11.18).—See B.P. 134,529; B., 1921, 74 A.

Fuel for use in internal-combustion engines. S. ISERMAN, W. VERNET, and E. Q. MOSES, ASSRS. to F. J. HALL and L. B. RATTERMAN (Re-issue 16,937, 17.4.28, of U.S.P. 1,654,259, 27.12.27).—See B., 1928, 222.

Purification of illuminating or like gas. HUMPHREYS & GLASGOW, LTD., ASSECS. of W. H. FULWEILER and C. W. JORDAN (B.P. 273,250, 26.3.27. U.S., 24.6.26).—See U.S.P. 1,632,758; B., 1927, 770.

Drying of gases. R. E. SLADE and V. E. PARKE, ASSRS. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,664,997,

3.4.28. Appl., 28.1.26. U.K., 28.1.25).—See B.P. 240,350; B., 1925, 957.

Production of stable [oil] emulsions. R. MEZGER (U.S.P. 1,665,105, 3.4.28. Appl., 1.4.27. Ger., 1.7.25).—See B.P. 254,701; B., 1927, 423.

Gas producers (B.P. 284,639).—See I. **Separation of sulphonic acids** (B.P. 284,859).—See III. **Filling cracks in brickwork** (B.P. 286,933). **Asphaltic or tarry emulsions** (B.P. 264,496).—See IX. **Insecticides** (B.P. 287,371).—See XVI. **Ammonium sulphoichthyolate** (U.S.P. 1,664,376). **Medicinal liquids** (U.S.P. 1,660,655).—See XX.

III.—ORGANIC INTERMEDIATES.

Synthesis of methyl alcohol from carbon monoxide and hydrogen. W. K. LEWIS and P. K. FROLICH (Ind. Eng. Chem., 1928, 20, 285—290).—Factors governing the catalytic synthesis of methyl alcohol are studied. Among the possible simple reactions between carbon monoxide and hydrogen the formation of methyl alcohol is accompanied by the largest decrease in volume, and hence is favoured by increase in pressure. Even with the best catalysts it is not possible to reduce the temperature of reaction below 300° , and whilst with a pressure of 1000 lb./in.² practically 100% methyl alcohol may be obtained at 300° , it is desirable to work at higher pressures to prevent the formation of methane. It has been found advantageous to use as a catalyst mixtures of two or more metallic oxides, of which one at least is non-reducible in carbon monoxide and hydrogen at atmospheric pressure at the temperature employed (e.g., oxides of copper, zinc, and aluminium). Owing to the exothermic character of the reaction (26,150 g.-cal. per mol. of gaseous methyl alcohol at 327°) the catalyst should be supported on a good heat conductor, e.g., granulated metallic copper, to prevent local overheating. The catalyst is prepared by precipitating the hydroxides by ammonia from a solution of the mixed nitrates at 85° , and is obtained as a gel into which the supporting copper is mixed prior to drying at 110° . It is sensitive to poisons, but by connecting several chambers in series it is possible to dispose of the catalyst poisons in the first chamber, thus protecting the contact material in the others. As the temperature is raised the yields of methyl alcohol pass through a maximum at 300 — 350° , and then fall off considerably owing to the decreased activity of the catalyst. Whilst the quantity formed per unit time increases with the rate of gas flow through the reaction chamber, the percentage conversion of carbon monoxide decreases owing to the shorter time of contact between the reacting gas and the catalyst. A pure alcohol is produced at about 3000 lb. pressure and at a relatively moderate temperature, and has a sp. gr. corresponding to 99—100% methyl alcohol. An apparatus suitable for the synthesis is described.

W. J. POWELL.

Application of the U.S.P. methyl alcohol test. W. L. O. WHALEY (Ind. Eng. Chem., 1928, 20, 320—322).—The method as given is suitable only for alcoholic solutions, hence when it is applied to flavours or medicinal preparations, the latter should be distilled and the distillates tested with the modified Schiff's reagent. It is important to prepare a sample of 5% total alcoholic

strength, as with stronger solutions the colour formation may be inhibited. The test is more sensitive if the sample is oxidised with dilute potassium permanganate and phosphoric acid, the excess of permanganate being decomposed with oxalic acid solution before distillation; under these conditions, however, many other substances yield similar colorations.

W. J. POWELL.

Use of *o*-cresol. BOVINI. **Cracking of hexadecane.** GAULT and SIGWALT.—See II.

PATENTS.

Production of formaldehyde by catalysis. E. A. BARBET (U.S.P. 1,661,063, 28.2.28. Appl., 23.10.23. Belg., 30.10.22).—In the oxidation of methyl alcohol to formaldehyde the air used is led in an inner tube through the catalyst chamber, and thus preheated is mixed with methyl alcohol vapour and passed to the catalyst.

T. S. WHEELER.

Manufacture of ethylene glycol [and ethylene oxide]. BRIT. DYESTUFFS CORP., LTD., K. H. SAUNDERS, and H. WIGNALL (B.P. 286,850, 8.2.27).—A mixture of about 40 pts. of ethylene chlorohydrin vapour and 60 pts. of steam, obtained for example by passing steam through a tower against a counter-current of crude chlorohydrin, is led up a packed tower against a downflowing stream of sodium carbonate solution. The glycol so produced dissolves in the salt solution and forms at the bottom of the tower a layer through which the chlorohydrin vapour and steam bubble. By a suitable heating of this layer, concentrated glycol may be drawn off through a constant-level trap, and its salt content allowed to crystallise out. If concentrated sodium hydroxide solution is used in place of sodium carbonate, ethylene oxide is produced and passes from the top of the tower to be dried and collected in the usual manner.

C. HOLLINS.

Manufacture of halogenated alcohols. I. G. FARBENIND. A.-G., and J. CALLEN (B.P. 286,797, 13.12.26. Addn. to B.P. 235,584; B., 1925, 738).—A secondary alcohol is used as solvent in place of the primary alcohol of the prior patent. Bromal, dissolved in *isopropyl* alcohol, is reduced by aluminium ethoxide at 115° in a stream or atmosphere of hydrogen to tribromoethyl alcohol, m.p. 80°.

C. HOLLINS.

Manufacture of primary aliphatic and cyclic amines. I. G. FARBENIND. A.-G. (B.P. 265,960, 7.2.27. Ger., 9.2.26).—Ketones, aldehydes, or their ammonia compounds are passed at 100–200° with hydrogen and ammonia over hydrogenation catalysts (reduced nickel and silica on pumice) preferably with the addition of dehydrating catalysts (aluminium phosphate, alumina) and activators (zinc oxide, magnesium oxide). *cyclo*-Hexylamine, *n*-butylamine, and *isobutylamine* are thus obtained together with only insignificant amounts of secondary and tertiary amines.

C. HOLLINS.

Concentration of aqueous solutions of fatty acids. F. E. LICHTENTHAELER (U.S.P. 1,660,756, 28.2.28. Appl., 21.1.26).—The dilute solution is treated with anhydrous aluminium ammonium sulphate (cf. U.S.P. 1,492,717; B., 1924, 671), ethyl alcohol, and an esterification catalyst, and the mixture is heated at about 90° to give ethyl acetate and the hydrated salt. The ester is recovered by distillation and hydrolysed to yield the concentrated acid.

T. S. WHEELER.

Manufacture of new aralkylated unsaturated fats and fatty acids and their sulphonic acids. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 286,796, 13.12.26).—Unsaturated aliphatic acids or fats are condensed with aralkyl halides, especially in presence of a catalyst such as zinc chloride, iron powder, aluminium chloride, to give compounds which are easily sulphonated, forming wetting-out or dispersing agents unaffected by acids or salt solutions. *E.g.*, oleic acid and rape oil are condensed with benzyl chloride in presence of zinc chloride, and sulphonated.

C. HOLLINS.

Manufacture of new derivatives from lignin-sulphonic acid (sulphite-cellulose waste). BRIT. DYESTUFFS CORP., LTD., and A. J. HALLWOOD (B.P. 286,808, 21.12.26).—Sulphite cellulose pitch is heated with aqueous ammonia at 110–120° in a closed vessel, to form an aminated derivative which is a highly-protective colloid suitable as a drying-down agent, and has tanning properties.

C. HOLLINS.

Preparation of vanillin and isovanillin. F. BOEDECKER (B.P. 285,156, and Addn. B.P. 285,551, [A] 12.11.26, [B] 6.11.26).—(A) The fission of the methylenedioxy-group of safrole or *isosafrole* by alcoholic alkali, followed by methylation with methyl sulphate and boiling with alcoholic hydrogen chloride, leads to a mixture of *isoeugenol* and *isochavibetol*, which may be oxidised in the usual manner to vanillin and isovanillin, separable by known means. Or the *isochavibetol* may be frozen out, methylated to 4-propenylveratrol (*isoeugenol* methyl ether), and converted by alcoholic alkali into a mixture of *isoeugenol* and *isochavibetol* from which the latter is again frozen out and added to the next batch for methylation. The *isoeugenol* is purified by crystallisation of its benzoyl or nitrobenzoyl derivative and subsequent hydrolysis; or it may be converted by way of its methyl ether into the mixture of *isoeugenol* and *isochavibetol*, and hence into *isovanillin*, if desired. (B) The mixture of *isoeugenol* and *isochavibetol* obtained is separated by taking advantage of the sparing solubility of sodium *isoeugenol*.

C. HOLLINS.

Manufacture of isoeugenol. SOC. ANON. PROD. CHIM. COVERLIN, Assees. of R. H. BOTS (B.P. 271,819, 2.2.27. U.S., 25.5.26).—Potassium eugenol is heated in aniline, *o*-toluidine, or other suitable amine at the b.p. to give *isoeugenol*, which may be oxidised to vanillin with nitrobenzene and alkali without isolation.

C. HOLLINS.

Separation of *m*-dinitrobenzene from its isomerides. H. J. WEILAND and I. GUBELMANN, Assrs. to NEWPORT CO. (U.S.P. 1,665,005, 3.4.28. Appl., 19.8.26).—Technical *m*-dinitrobenzene is treated in the presence of water with an alkali-forming substance, which converts the *o*- and *p*-isomerides into water-soluble derivatives without decomposing the *m*-dinitrobenzene. The latter may then be separated in a pure condition.

F. G. CLARKE.

Alkylation of carbazole. R. W. HESS and J. C. SIEMANN, Assrs. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,662,061, 13.3.28. Appl., 25.1.22).—*N*-Alkyl derivatives of carbazole are produced by the interaction of an alkylating agent with carbazole in the presence of an acid-binding agent.

F. G. CLARKE.

Manufacture of indophenols. R. W. HESS and O. D. CUNNINGHAM, Assrs. to NAT. ANILINE AND CHEM. CO., INC. (U.S.P. 1,662,062, 13.3.28. Appl., 25.3.22).—An indophenol is produced by condensing carbazole or a derivative thereof with excess of *p*-nitrosophenol in the presence of sulphuric acid. With *N*-carbazole derivatives the reaction is commenced at -5° and then maintained below 0° . F. G. CLARKE.

Manufacture of new quaternary ammonium compounds from halogen-substituted tertiary aromatic amines. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 286,984, 10.12.26).—Alkylating agents are obtained by interaction of alkyl sulphates or alkyl arylsulphonates and halogenated tertiary amines of the benzene or naphthalene series. 3-*Chloro-p*-tolyltrimethylammonium *p*-toluenesulphonate, m.p. 154° , 1-*chloro-β*-naphthyltrimethylammonium methyl sulphate, m.p. 98° , *o*-chloro- (with 2:4-dichloro-)phenyltrimethylammonium methyl sulphate, and the methosulphates of 3:3'-dichloro- [m.p. 218° (decomp.)] and 3:3'-dibromo-4:4'-tetramethyldiaminodiphenylmethanes are described. C. HOLLINS.

Separation of mono- and di-alkyl derivatives of aromatic amines. SILESIA VER. CHEM. FABR. (B.P. 280,877, 21.10.27. Ger., 19.11.26).—The mixture of secondary and tertiary amines is treated with a slight excess of phthalic anhydride in an inert solvent (benzene etc.). The phthalamic acid formed from the secondary amine dissolves in the benzene, from which it is removed by shaking with aqueous alkali and hydrolysed with boiling acid; the tertiary amine is recovered from the benzene solution by distillation. C. HOLLINS.

Preparation of disulphide compounds. GOODYEAR TIRE & RUBBER Co., Asses. of C. McK. CARSON (B.P. 277,338, 6.9.27. U.S., 7.9.26).—Mercaptans (in particular, 1-mercaptobenzthiazole) are converted into corresponding disulphides by treatment in a solvent (e.g., carbon tetrachloride) with sulphur chloride, sulphur and hydrogen chloride being produced. The disulphide, m.p. 175° , from 1-mercaptobenzthiazole is a vulcanisation accelerator. C. HOLLINS.

Separation and purification of sulphonic acids of high mol. wt. G. PETROFF (B.P. 284,859, 1.2.27).—Sulphonated oils and other sulphonic acids of high mol. wt. (sulphonated black naphtha, octahydroanthracene-sulphonic acid, Twitchell's reagents, etc.) are mixed with cellulosic material (sawdust, wood-powder, hydrocellulose), dried, and extracted successively with mineral oil (benzine), benzene, and alcohol. The acids are recovered from the extracts by washing with water, aqueous alcohol, or alcohol, or by conversion into salts. C. HOLLINS.

Manufacture of *o*-aminoaldehydes and *o*-amino ketones of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 267,163, 8.3.27. Ger., 8.3.26).—The anthraquinoneoxazoles obtained by the action of fuming sulphuric acid on 1-nitro-2-alkylantraquinones (B.P. 147,001; B., 1922, 50 A) are reduced with ammonia and sodium hyposulphite, or with ferrous sulphate, to 1-aminoanthraquinone-2-aldehydes or corresponding ketones. The oxazole reaction mixture may be diluted and reduced with ferrous sulphate without

isolation. By these two reactions 1-nitro-2-methylanthraquinone is converted into 1-aminoanthraquinone-2-aldehyde, m.p. 239° , 1-nitro-2-ethylanthraquinone into 1-aminoanthraquinone-2-methyl ketone, m.p. 220° , 1:5-dinitro-2-methylanthraquinone into 5-nitro-1-aminoanthraquinone-2-aldehyde, m.p. 228° , 1:5-dinitro-2:6-dimethylanthraquinone into 1:5-diamino-2:6-dialdehydeanthraquinone, m.p. 340° , and 1:8-dinitro-2:7-dimethylanthraquinone into the 1:8:2:7-compound. C. HOLLINS.

Manufacture of *o*-aminocarboxylic acids of the anthraquinone series and substitution products thereof. I. G. FARBENIND. A.-G. (B.P. 267,164, 8.3.27. Ger., 8.3.26).—Anthraquinoneoxazoles (B.P. 147,001; B., 1922, 50 A) are converted by hot dilute alkalis into 1-aminoanthraquinone-2-carboxylic acids. The preparation of 1-aminoanthraquinone-2-carboxylic acid, its 5-nitro-derivative, m.p. 325° , 1:5-diaminoanthraquinone-2:6-dicarboxylic acid, and the 1:8:2:7-isomeride, is described. C. HOLLINS.

Manufacture of polycyclic compounds containing a carbonyl group. I. G. FARBENIND. A.-G. (B.P. 263,163, 15.12.26. Ger., 15.12.25).—Aromatic and heterocyclic ketones containing halogen in positions *ortho* or *peri* to the carbonyl group are cyclised by heating with a slightly alkaline salt or acid-binding agent (sodium carbonate, calcium carbonate, sodium acetate, magnesium oxide), especially in presence of catalysts (copper, copper carbonate, iron carbonate). C. HOLLINS.

Manufacture of catalysts and their application in the reduction or hydrogenation of organic compounds. I. G. FARBENIND. A.-G. (F.P. 621,434, 20.7.26. Ger., 22.7.25).—Further to B.P. 255,884 (B., 1927, 528) are the following. For the preparation of a nickel catalyst, dust-free air is passed for 4 hrs. at 70° through aqueous nickel nitrate solution containing 25% ammonia and finely divided silica gel, the precipitate being dried and reduced in hydrogen first at 100 – 130° and then at 550° ; the catalyst is finally cooled in hydrogen or nitrogen and transferred to the liquid (water, pyridine, decalene) for use. The precipitate obtained by adding nickel nitrate solution at 70° dropwise to a very dilute sodium carbonate solution may be similarly worked up. These catalysts are used in the reduction of various nitro-compounds with hydrogen at 60 – 100° to give amines and diamines, namely, α -naphthylamine, *m*- and *p*-phenylenediamines, cresidine, *o*-chloroaniline, 4-chloro- α -naphthylamine (m.p. 95°), aniline-*o*-sulphonic acid, *o*-aminoacetanilide (m.p. 120 – 121° : hydrochloride, m.p. 132°), *p*-aminoacetanilide, *p*-aminobenzoic acid, ethyl 3-amino-4-hydroxybenzoate, naphthastyril and 5-amino-1-naphthoic acid (from 1:5[8]-nitronaphthoic acid), 2:5-dichloroaniline (m.p. 50°), 2:4:6-triaminotriphenyl-1:3:5-triazine, 5-aminoacacenaphthene, methyl 5-*m*-nitrobenzoylsalicylate (m.p. 130 – 131°), 3-amino-*p*-tolyl carbonate (m.p. 146 – 147°), 4-chloro-2-aminophenol; benzeneazosalicylic acid gives 5-aminosalicylic acid, and azoxybenzene yields aniline. Reduction of nitriles under these conditions gives a larger proportion of primary and less secondary amine. From phenylacetoneitrile are obtained 63% of β -phenylethylamine and 18% of secondary base; from *p*-nitrophenylacetoneitrile *p*-aminophenylacetoneitrile and

β -*p*-aminophenylethylamine, b.p. about 147°/11 mm., result at 50 and 90°, respectively. Similarly α -naphtho-nitrile gives (1-naphthylmethyl)amine (70%) and di-(1-naphthylmethyl)amine (20%), acetonitrile ethylamine, and propionitrile *n*-propylamine (80%). Other reductions described are: crotonaldehyde to *n*-butaldehyde and thence to *n*-butyl alcohol; cinnamonnitrile to β -phenylpropionitrile and thence to γ -phenyl-*n*-butylamine; acetophenone to phenylmethylcarbinol and thence to ethylbenzene; diethyl ketone to diethylcarbinol (γ -hydroxypentane); ethyl acetoacetate to ethyl γ -hydroxybutyrate; 2-*p*-toluoylbenzoic acid to 4-methyldiphenylmethane-2'-carboxylic acid, and the nitro-compound to 3-amino-4-methyldiphenylmethane-2'-carboxylic acid, m.p. 130—136°; benzophenone to diphenylmethane, b.p. 260—262°; *m*-nitro-*p*-tolyl sulphonyl- α -naphthyl ketone to *m*-amino-*p*-tolylsulphonyl-1-naphthylmethane; 2:2'-dinitrodi-*p*-anisyl ketone to 2:2'-diaminodi-*p*-anisylmethane, m.p. 110°; phenyl *m*-nitro-*p*-tolyl ketone to 3-amino-4-methyldiphenylmethane (hydrochloride, m.p. 218°); 3-nitro-di-*p*-tolyl ketone to 3-amino-di-*p*-tolylmethane (hydrochloride, m.p. 210—211°); acetoxime to isopropylamine; quinaldine to tetrahydroquinaldine at 80°, or decahydroquinaldine at 130°; pyridines to piperidines.

C. HOLLINS.

Manufacture of carboxylic acids of acenaphthene. I. G. FARBENIND. A.-G. (B.P. 274,894, 22.7.27. Ger., 24.7.26).—The mono- or di-acyl derivatives, obtained by condensing acenaphthenes with acetyl or chloroacetyl chloride in presence of aluminium chloride, are oxidised with alkaline hypochlorite to mono- or dicarboxylic acids (and chloroform). The methylene groups of the *peri*-ring are not attacked. The 5-carboxylic acid, m.p. 217°, 5:6(?)-dicarboxylic acid (from bischloroacetylacenaphthene, m.p. 180°), and 5-bromo-6-carboxylic acid (from the bromination product, m.p. 164°, of 5-acetylacenaphthene), are described.

C. HOLLINS.

Method of sublimation. C. FIELD, Asst. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,662,056, 13.3.28. Appl., 22.3.20).—Substances such as anthracene or anthraquinone in a finely-divided condition are charged into a preheated receptacle and stirred until they are plastic. A heated gas or vapour is then introduced to promote sublimation.

F. G. CLARKE.

Manufacture of paraformaldehyde. E. KUSS, Asst. to I. G. FARBENIND. A.-G. (U.S.P. 1,666,708, 17.4.28. Appl., 18.6.26. Ger., 24.6.25).—See B.P. 267,768; B., 1927, 428.

Production of vinyl acetate. E. BAUM, H. DEUTSCH, W. O. HERRMANN, and M. MUGDAN, Assrs. to CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (U.S.P. 1,666,482, 17.4.28. Appl., 15.6.22. Ger., 23.6.21).—See B.P. 182,112; B., 1923, 861 A.

Stearic acid (U.S.P. 1,659,790).—See XII. **Pyridine derivatives** (B.P. 259,982 and 265,167).—See XX.

IV.—DYESTUFFS.

Lake pigments. WAGNER and others.—See XIII.

PATENTS.

Manufacture of new vat dyes of the anthraquinone series. W. CARPMAEL. From I. G. FARB-

ENIND. A.-G. (B.P. 285,555, 16.11.26).—A 3-halogenopyrazolanthrones, particularly the 3-bromo-compound prepared from 3-bromo-1-aminoanthraquinone by diazotisation, reduction, and cyclisation, is heated with an acid-binding agent (potassium acetate) and a catalyst (copper acetate) in boiling nitrobenzene to give a blue vat dye.

C. HOLLINS.

Manufacture of vat dyes of the benzanthrone series. I. G. FARBENIND. A.-G. (B.P. 263,200, 20.12.26. Ger., 18.12.25. Addn. to B.P. 242,620; B., 1927, 101).—The sulfoxides or sulphones obtained by oxidising 3-benzanthronyl thioethers, e.g., with persulphates, hydrogen peroxide, or nitric acid (the last also introduces nitrogen), give vat dyes when treated with alcoholic alkali. 3-Benzanthronyl methyl sulphide thus gives a violet vat dye. 3:3'-Dibenzanthronyl sulphide is oxidised and dinitrated by mixed acid, and the diamino-3:3'-dibenzanthronyl sulphone obtained by subsequent reduction yields with alcoholic alkali a violet-blue vat dye.

C. HOLLINS.

Manufacture of condensation products of the anthracene series. I. G. FARBENIND. A.-G., Asses. of MEISTER, LUCIUS, & BRÜNING (B.P. 261,400, 12.11.26. Ger. 13.11.25).—Anthranols, oxanthranols, or anthrones are condensed with crotonaldehyde in presence of sulphuric acid in the absence of an oxidising agent; e.g., anthrone gives 1-methylbenzanthrone, m.p. 113—114° (cf. B.P. 244,120; B., 1927, 326).

C. HOLLINS.

Manufacture of derivatives of naphthaquinones. R. LANTZ and A. WAHL, Assrs. to SOC. ANON. DES MAT. COL. & PROD. CHIM. DE ST.-DENIS (U.S.P. 1,666,241, 17.4.28. Appl., 21.1.26. Fr., 21.1.25).—See B.P. 246,482; B., 1927, 39.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Transmission of ultra-violet radiation by various fabrics. H. R. HIRST, P. E. KING, and P. N. LAMBERT (J. Soc. Dyers and Col., 1928, 44, 109—113).—Samples of various fabrics (wool, silk, cotton, and artificial silks) were exposed to the radiation from a mercury arc filtered through a screen of Chance's ultra-violet glass. All fabrics cut off a large proportion of the radiation, the chief controlling factors being the weave and texture of the material, and not the kind of fibre of which it is composed. For equal thicknesses of material, wool has generally the highest transmissive power, whilst dyeing diminishes this power in the case of all fabrics.

B. P. RIDGE.

Cleaning of fur and leather garments. M. H. GOLDMAN and C. C. HUBBARD (U.S. Bur. Stand. Tech. Paper, 360, 1927, 22, 183—197).—Fur and leather goods retain their colour, appearance, and pliability during dry-cleaning if a small percentage (1.25% for leather and 2.5% for furs) of paraffin wax, m.p. 45°, is added to the naphtha. If, as has been found the case in works' practice, it is undesirable to add the paraffin wax to the cleaning liquor, the material, after cleaning in the usual way, may be soaked in the paraffin wax-naphtha mixture for about 5 min., centrifuged, and exposed to warm air at a temperature not above 50°. A standard practice for cleaning fur and leather materials is suggested.

D. J. NORMAN.

Viscose. XIII and XIV. G. KITA, R. TOMIHISA, K. NAKAHASHI, and J. ONOHARA (J. Cellulose Inst. Tokyo, 1928, 4, 25—30; 31—34).—By the addition of dextrose, viscose may be spun after a longer ripening than when ammonium sulphate is used in the bath, whilst increasing the quantity of dextrose slightly shortens the necessary time of ripening. Over-ripe viscose cannot be spun from a bath containing a large quantity of dextrose, the action of the latter being affected by the degree of ripening. For viscose spun from such a bath the tensile strength of the fibres is increased but the extensibility is diminished. In a bath containing 8% of sulphuric acid a less ripe viscose may be spun than in one containing much sodium sulphate, whilst if 12% of sulphuric acid is used the opposite is the case. Sodium sulphate has no effect on the tensile strength and extensibility of the fibres. No special influence is exerted by magnesium sulphate of 6% concentration, whilst with under-ripe viscose, in the initial stages of spinning, a 12% solution increases the tensile strength.

Fibres from a bath containing zinc sulphate are at first coloured yellow, but afterwards decolorised. When 1—3% of the salt is present a less ripe viscose may be spun than when no zinc sulphate is added, but for a 5% solution the opposite holds, *i.e.*, a less ripe viscose is more difficult to spin than when the salt is absent. The influence of zinc sulphate on the properties of the fibres depends on the properties of the viscose, but with a moderately ripened viscose fibres of increased extensibility and tensile strength may be obtained.

B. P. RIDGE.

Decay of oak-wood. A. BRANDL (Brennstoff-Chem., 1928, 9, 89—94).—Examination of samples of decayed oak-wood by Odén's alkaline-extraction method (B., 1926, 82, 568) or by the acetyl bromide process (Grosskopf, B., 1926, 939) shows that much of the decayed wood is already humified, a further proportion being converted into decomposition products of lignin, as lignic acid. Values for lignin determined by the sulphurous acid method are about 5% high. The soluble matter (%) extracted from decayed (young) wood from the bark of the tree and from older wood from the trunk by means of cold water, hot water, 5% sodium carbonate solution, 5% sodium hydroxide solution, and 10% sodium hydroxide solutions, respectively, are 4.5, 4.8; 17.1, 10.0; 29.5, 33.1; 25.1, 26.0; 7.3, 3.4; these woods contained 9.3% and 6.9% of cellulose, respectively, and 16.5% and 22.7% of insoluble residue. Precipitation of the sodium hydroxide-soluble fraction with alcohol affords a humic acid closely resembling that obtained by the acetyl bromide process, the chief difference being in the high methoxy content, which is due to absorbed alcohol. The carbonyl content is a little higher than that of the original decayed wood (2.66—2.68; methoxyl 10.95). The calcium salt has equivalent 318, corresponding with that of the humic acid isolated by Odén from coal. Nitration of the humic acid affords a product (impure) containing 47.81% C, 3.73% H, 3.99% N, and closely resembling the corresponding product from the humic acid of coal, together with a yellow "nitrophenolic" substance, 42.38% C, 2.53% H, resembling that described by Tropsch and Schellenberg

(B., 1924, 323). The fraction soluble in sodium hydroxide and in alcohol (59.84% C, 4.76% H) has acid value 70.73 (cold), 88.86 (hot), carbonyl value 2.63, methoxyl value 11.34 (due to absorbed alcohol), and is probably a hymatomelanic acid, contaminated with tannins and other soluble products. It decomposes slowly on drying at 70—80°, more rapidly at 105—110° (cf. Eller and Schöppach, B., 1926, 257). The heats of combustion of the products isolated by the alkaline extraction process fall fairly regularly with the carbon content. The methoxy-content of the products tends to decrease, the carbonyl value to increase as compared with the original decayed wood, and the ease with which these products undergo oxidation or condensation and polymerisation is attributed to the presence of ketonic or aldehydic groups. These results indicate that a part of the lignin has already been converted into humic acid in the decayed wood, and support the view advanced by Fischer and Schröder that coal is essentially a decomposition product of lignin.

R. BRIGHTMAN.

Position of researches on [the constitution of] lignin. K. G. JONAS (Papier-Fabr., 1928, 26, 221—229).—A review of the constitutional formulae ascribed to lignin by various authors. The formula proposed by Klason (A., 1923, i, 187), which represents lignin as a condensation product of coniferylaldehyde, is rejected on the grounds that it does not explain why one of the two methoxyl groups is very readily hydrolysed whereas the other is difficult to attack; that a substance so constituted should yield large quantities of aromatic substances on oxidation, whereas only very small yields of such products are obtainable from lignin; and that it is not in accordance with the fact that most analyses of lignin indicate the presence of oxygen atoms (usually 3 per 20 carbon atoms) other than those in hydroxyl, alkoxy, and carboxyl groups. The author favours the formula of Schrauth (A., 1923, i, 443) and regards lignin as a complex structure which contains tetrahydrofuran and cyclohexenol rings and is derived from a hexose by dehydration and subsequent reduction. The formula proposed—a modification of Schrauth's—explains more satisfactorily the difference in the rates of hydrolysis of the two methoxyl groups and includes an acetyl group which has been shown to be present in natural lignin.

W. J. POWELL.

Edible cellulose. HARDING.—See XIX.

PATENTS.

Protection of animal fibres etc. against the effects of alkaline or acid media. BRIT. DYESTUFFS CORP., LTD., R. S. HORSFALL, and L. G. LAURIE (B.P. 285,554, 16.11.26).—A soluble condensation product of carbamide with an aldehyde, *e.g.*, dihydroxydimethyl-carbamide [dimethylolurea], is added to acid or alkaline baths to protect wool, silk, fur, etc. from injury in the bath. Examples are the scouring of wool, dyeing silk with vat dyes, "killing" furs, dyeing worsted with acid colours, and mordanting wool.

C. HOLLINS.

Sizing of fibres. G. M. CLARK. From AGASOTE MILLBOARD Co. (B.P. 286,948, 2.7.27).—A more efficient and even distribution of the sizing agent over the surface of the fibres is obtained if tannic acid is added to the

beater before precipitation of the size by the addition of alum. Thus, in the manufacture of waterproof pulp boards, the stock may be sized with 11–13% of sodium linoleate to which has been added 10–20% of its weight of tannic acid, preferably in the form of a commercial tanning extract. D. J. NORMAN.

Manufacture of waterproof compositions. C. G. A. LUNDBERG (B.P. 271,076, 10.5.27. Swed., 11.5.26).—Roved wool or cotton, or waste material from wool and cotton manufacture, is first purified, e.g., by treatment with dilute sulphuric acid for about 12 hrs., and, after washing and drying, is impregnated at about 80° for, e.g., 30 min. with an acid solution containing a zinc salt, e.g., a solution containing 78 pts. of zinc chloride, 14 pts. of zinc sulphate, and 8 pts. of nitric acid, which has been boiled for about 2 hrs. before use. The treated material, after removing the excess of liquor, is dried, disintegrated, and mixed, optionally after the addition of fillers, with a rubber solution prepared by adding a solution of waste rubber in linseed oil to molten asphalt, rosin, etc. The resulting mixture is moulded under heat and pressure and is finally vulcanised. D. J. NORMAN.

Method of laundering. J. TAKAMINE and J. TAKAMINE, JUN., ASSRS. to TAKAMINE FERMENT CO. (U.S.P. 1,660,458, 28.2.28. Appl., 17.12.21).—Starch, gums, oils, etc. are removed from the goods by treatment with tepid water containing diastatic and proteolytic enzymes derived by extraction of a vegetable fungus, e.g., *Aspergillus oryzae*. T. S. WHEELER.

Production of cellulose and other chemical products. H. WALLIN (U.S.P. 1,660,696, 28.2.28. Appl., 28.9.25. Sweden, 8.10.24).—In the manufacture of cellulose by cooking wood with an alkali silicate solution, the spent liquor is regenerated by evaporation and calcination of the residue at 700°, sodium carbonate and silicic acid being added to make up loss of alkali silicate. T. S. WHEELER.

Opening-up of materials containing cellulose. I. G. FARBERIND. A.-G. (B.P. 279,036, 26.8.27. Ger., 13.10.26. Addn. to B.P. 274,892; B., 1928, 154).—The material, e.g., moist fir wood, is treated for one or more hours with a mixture of air and oxides of nitrogen at 40–50°, and is then exposed to the action of air alone for about 24 hrs. at the ordinary temperature or at about 50°. D. J. NORMAN.

Treatment of celluloid scrap. RHEINISCHE GUMMI- U. CELLULOID-FABR. (B.P. 277,626, 9.7.27. Ger., 18.9.26).—The comminuted or pulverised material is intimately mixed with, e.g., 2% of a non-volatile solvent for nitro-cellulose (triphenyl phosphate, ethyl phthalate, acetanilide, ethylacetanilide, ethyl-*p*-toluenesulphonamide) and is then moulded under heat and pressure. The moulding may be done at relatively low temperatures and the resulting products are homogeneous and of but slightly reduced hardness. D. J. NORMAN.

Manufacture of gut strings. T. SARRA (U.S.P. 1,664,323, 27.3.28. Appl., 22.3.26).—Bovine intestines are treated with caustic alkali solution to separate the inner and outer layers; the inner layer, after further treatment with caustic alkali solution to strengthen it, is then twisted into gut strings. D. J. NORMAN.

Sizing of paper. J. A. DE CEW (U.S.P. 1,663,976, 27.3.28. Appl., 22.9.26).—The rosin size is precipitated with aluminium sulphate that has been dissolved in water and made basic. D. J. NORMAN.

Production of permanent-finish vegetable [fibrous] material. Vegetable yarn and fabric with wool-like effects. A. BODMER, ASSR. to HEBERLEIN PATENT CORP. (U.S.P. 1,666,082–3, 17.4.28. Appl., [A] 11.4.23, [B] 12.4.23. Ger., 15.4.22).—See B.P. 196,298; B., 1924, 332.

Production of artificial silk. W. H. GLOVER and C. F. TOPHAM, ASSRS. to COURTAULDS, LTD. (U.S.P. 1,665,958, 10.4.28. Appl., 29.10.26. U.K., 7.1.26).—See B.P. 268,455; B., 1927, 472.

Production of artificial silk filaments by drawing. K. GRUNERT (U.S.P. 1,665,453, 10.4.28. Appl., 4.10.26. Ger., 8.10.25).—See B.P. 263,462; B., 1927, 138.

After-treatment of freshly-spun artificial silk. F. J. GAHLERT (U.S.P. 1,666,090, 17.4.28. Appl., 28.7.26. Ger., 9.6.25).—See B.P. 283,752; B., 1928, 228.

Production of cellulose by decomposition of vegetable fibres. O. C. STRECKER (B.P. 284,846, 13.1.27).—See U.S.P. 1,658,213; B., 1928, 295.

Hydrolysis of cellulosic substances. SOC. DES BREVETS ÉTRANGERS LEFRANC & CIE. (B.P. 271,410, 11.2.27. Fr., 20.5.26).—See F.P. 616,424; B., 1928, 187.

Manufacture of a cellulose-derivative film. H. J. HANDS, ASSR. to SPICERS, LTD. (U.S.P. 1,666,377, 17.4.28. Appl., 25.1.27. U.K., 24.9.26).—See B.P. 281,803; B., 1928, 187.

Stencil sheet. S. HORII (U.S.P. 1,664,777, 3.4.28. Appl., 14.1.27).—See B.P. 250,798; B., 1926, 534.

Manufacture of a composite adhesive [for sand paper]. F. G. OKIE, ASSR. to MINNESOTA MINING & MANUF. CO. (U.S.P. 1,659,611, 21.2.28. Appl., 4.3.22).—See B.P. 219,341; B., 1924, 905.

Machines for [stretch-spinning] manufacture of artificial textile threads etc. H. KINDERMANN (B.P. 287,602, 23.11.26).

Derivatives from ligninsulphonic acid (B.P. 286,808).—See III.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Colouring of leather. LAMB.—See XV. **Dye absorption of soils.** CROUCHER.—See XVI.

PATENTS.

Dyeing, printing, or stencilling of materials composed of or containing cellulose ethers. H. DREYFUS (B.P. 285,104, 8.10.26).—The methods of the prior patents for the dyeing etc. of acetate silk are extended to the dyeing etc. of cellulose ethers, such as methyl-, ethyl-, and benzyl-celluloses. C. HOLLINS.

Dyeing, printing, and stencilling of materials made with or containing cellulose derivatives. BRIT. CELANESE, LTD., and H. C. OLPIN (B.P. 285,641, 9.2.27).—Cellulose esters and ethers, including immunised

cotton, are dyed etc. by means of β -sulphatoethyl derivatives of aminoanthraquinones. 1- β -Sulphatoethyl-aminoanthraquinone gives a scarlet, and the corresponding derivatives of 1-amino-2-methyl- (scarlet), 1-amino-4-hydroxy- (violet), 1:4-diamino- (mono-, blue-violet; di-, pure blue), 2-bromo-1:4-diamino- (mono-, blue-violet), 1-amino-4-methylamino- (pure blue), and 1:5-diamino- (di-, bluish-red) -anthraquinones are also described.

C. HOLLINS.

Dyeing of fur. A. WINOGRADOFF, Assr. to INECTO, INC. (U.S.P. 1,660,826, 28.2.28. Appl., 1.4.26).—The fur is treated with dilute silver nitrate solution, and, after exposure to light, is immersed in *p*-phenylenediamine solution to develop opalescent colours. T. S. WHEELER.

Toning colours in fabrics. I. B. FUNK (U.S.P. 1,659,598, 21.2.28. Appl., 15.9.24).—A coloured fabric comprising a wool nap and a cotton warp is treated with a paste of diatomaceous earth and dilute sulphuric acid. The capillary attraction of the nap removes the acid from the paste, and the warp is not attacked.

T. S. WHEELER.

Treatment of printed or dyed fabrics. R. W. JACOBY, Assr. to DELTEX Co. (U.S.P. 1,663,845—6, 27.3.28. Appl., [A] 3.5.26, [B] 13.8.27).—(A) In the ageing or reduction of printed or dyed fabrics, moisture is supplied in the vicinity of the exothermic reactions so that its evaporation absorbs heat and maintains the heat-carrying medium as a saturated vapour. (B) Suitable apparatus is described.

F. G. CLARKE.

Colour printing on textile fabrics. SOC. ANON. DES ÉTABL. PETITDIDIER (ANC. MAISON JOLLY-BELIN) (B.P. 261,399, 12.11.26. Fr., 12.11.25).—Immunised cotton, with or without viscose silk, with a backing of cotton, wool, silk, etc. is printed with a cellulose acetate dye, or an acid dye having affinity for immunised cotton, or a basic dye in conjunction with tannin or acetanol, and steamed to fix the colour on the immunised cotton. The viscose and the backing are then discharged to white by rinsing with water followed by 0.1% sodium hyposulphite at 75°. The last traces of colour may be removed from a silk backing by means of dilute acid.

C. HOLLINS.

Production of pattern effects in fabrics. H. MÜLLER, Assr. to A.-G. CILANDER (U.S.P. 1,660,042, 21.2.28. Appl., 25.2.26. Ger., 20.1.26).—A mixed fabric containing animal and vegetable fibres is printed with an alkaline paste and steamed to destroy the animal fibres.

T. S. WHEELER.

Dyeing preparation for cellulose esters. R. METZGER, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,666,715, 17.4.28. Appl., 4.3.26. Ger., 7.5.25).—See B.P. 271,550; B., 1927, 553.

Preparation of colour pastes for colour printing. M. FREIBERGER and A. HOLTMANN (U.S.P. 1,665,162, 3.4.28. Appl., 25.10.24. Ger., 14.12.23).—See B.P. 226,164; B., 1925, 879.

[Machines for] the fluid treatment of artificial silk yarns. J. BRANDWOOD (B.P. 287,731, 19.4.27).

Protection of animal fibres (B.P. 285,554).—See V. **Coloured rubber** (U.S.P. 1,663,436).—See XIV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Determination of the caustic value of lime. Two **filtration devices.** W. M. SHAW, W. H. MACINTYRE, and J. E. UNDERWOOD (Ind. Eng. Chem., 1928, 20, 312—314).—The slow settling of magnesia suspensions in sucrose solution renders the following method of filtration without contact with carbon dioxide preferable in the analysis of magnesian limes. The ground lime is shaken with sugar solution in a 500 c.c. Erlenmeyer flask, the stopper of which carries a soda-lime tube and a siphon connexion. To the latter and within the flask is connected a filter cone made from a cut-down pipette and packed with cotton covered by macerated filter paper. Aliquot parts are drawn for titration by the siphon. Any moisture in the filter is removed by the first aliquot. Another type of apparatus consists of a flask with protected air connexion and measuring pipette with 3-way stopcock at each end. Connected to the latter and within the flask is an alundum filtering thimble. The filtrate is drawn up into the pipette by suction (cf. following abstract). C. IRWIN.

Effects of certain impurities on the determination of causticity of limes by modifications of the sugar and Scaife methods. W. M. SHAW and W. H. MACINTYRE (Ind. Eng. Chem., 1928, 20, 315—319).—The filtration devices described in the preceding abstract were used in this investigation. Magnesia carefully freed from lime was without effect on the determination of caustic lime with sugar, but lowered the value found by the Scaife method. Added silica decreased the values found in both cases, no doubt by silicate formation, but greater decreases occurred in the sugar method. If a mixture of lime, magnesia, and silica is calcined, some of the silica always passes into combination with lime. A similar result was obtained by igniting natural magnesium silicate (serpentine) with lime. Addition of calcium silicate was without effect in the time required for determination, but reduced causticity in 24 hrs.' contact. Alumina also causes some reduction in causticity, whilst added calcium aluminate increases it by hydrolysis.

C. IRWIN.

Determination of free alkali in hypohalogenite solutions. E. RUPP and F. LEWY (Z. anal. Chem., 1928, 73, 283).—The determination of free alkali in solutions of hypochlorite or hypobromite is carried out by titration with standard acid in the presence of methyl-orange after first destroying the hypohalogenite by addition of hydrogen peroxide.

A. R. POWELL.

Determination of small amounts of carbon monoxide in air. G. M. EDELL (Ind. Eng. Chem., 1928, 20, 275).—Samples of air containing known amounts of carbon monoxide from 0.001% to 0.06% were analysed by the iodine pentoxide method. It was found that only the purest iodine pentoxide could be used. Even then with pure air a constant though small liberation of iodine occurred, but it could be corrected for and the results obtained were of fair accuracy.

C. IRWIN.

Ammoniacal nitrogen of peats and humus soils. ELLIS and MORISON. **Dusting mixtures.** SWINGLE.—

See XVI. **Fumigation.** BUTTENBERG and GAHRTZ.—
See XXIII.

PATENTS.

Manufacture of sulphuric acid. SOC. GÉN. MÉTALL. DE HOBOKEN (B.P. 284,208, 16.12.27. Fr., 24.6.27).—The consumption of nitric acid is reduced when using cold sulphurous gases to manufacture sulphuric acid having d 1.7 or less by introducing the cold gases into the plant beyond the denitration apparatus, the denitration of the acid being effected by hot sulphurous gases in a tower filled with irregularly-shaped filling material of smaller diameter than 40 mm. W. G. CAREY.

Manufacture of sulphuric acid. A. SHARP. From SOC. GÉN. MÉTALLURGIQUE DE HOBOKEN (B.P. 286,865 and 286,980, [A] 19.2.27, [B] 23.8.27).—(A) When manufacturing sulphuric acid from sulphur dioxide of varying temperature and concentration a single reaction chamber is used of such shape that maximum volume is obtained for minimum surface and is filled with packing having a high heat capacity, the temperature being kept constant by varying the temperature of the circulating acid. (B) Gases with a varying sulphur dioxide content are first treated by any known contact process and the remaining sulphurous gases are treated in towers, which are rinsed with nitrosylsulphuric acid. W. G. CAREY.

Preparation of vanadic acid. H. G. C. FAIRWEATHER. From SELDEN Co. (B.P. 287,401, 14.9.27).—Impure alkali vanadate solutions are treated with alkali carbonates and with oxidising agents, such as chlorine, bromine, or peroxides, to convert all the vanadium into the quinquivalent state and to precipitate metals which form insoluble carbonates and peroxides. If arsenic and phosphoric acids are present magnesium chloride is added to the acidified filtrate and the solution is further oxidised with nitric acid or a halogen, made just ammoniacal, and again filtered after 8–10 hrs. The precipitate contains arsenic, phosphorus, iron, and silica; the filtrate is heated to boiling, saturated with ammonium chloride, and cooled, and the ammonium metavanadate which crystallises out is collected, washed, and ignited at 200–300° in air, then at 300–400° in oxygen to obtain pure vanadium pentoxide free from lower oxides. A. R. POWELL.

Drying, neutralising, and delivering ammonium salts and other granular substances. INTERNAT. CEMENT-GUN Co., G.M.B.H., Assees. of F. BINSWANGER (B.P. 277,652, 8.9.27. Ger., 14.9.26).—The substance is fed into a drying chamber having superposed trays and is caused to fall from tray to tray by rotating, gradually descending scrapers; heated air mixed with neutralising gases if necessary is passed upward in a zig-zag path. The dried material is thrown by centrifugal action on to the rim of a rotating disc and is discharged by an air blast. W. G. CAREY.

Magnesia [from dolomite]. Refractory magnesia compounds. W. KOEHLER (U.S.P. 1,664,630–1, 3.4.28. Appl., [A] 21.12.23, [B] 15.3.26).—(A) A mixture of dolomite and magnesium chloride is calcined to obtain magnesia, calcium chloride, and carbon dioxide. The calcium chloride is leached out, the solution treated with the oxides obtained by calcining a further quantity of

dolomite alone and saturated with carbon dioxide, and the magnesium chloride thus regenerated separated from the calcium carbonate precipitate and used again in the process. (B) Finely-divided raw dolomite is treated with magnesium chloride equivalent to its lime content, the mixture heated until it is dehydrated, and the calcium chloride leached out. The residual magnesia is calcined to obtain a refractory. A. R. POWELL.

Production of high-grade magnesium oxide. W. KOEHLER (U.S.P. 1,661,043, 28.2.28. Appl., 15.10.27).—Dolomite is heated with magnesium chloride at 700° to yield a mixture of calcium chloride and magnesium oxide which is extracted with water. T. S. WHEELER.

Recovery of zinc from solutions [of zinc salts]. S. C. SMITH (B.P. 287,186, 18.9.26).—Purified zinc chloride or sulphate solutions obtained from the leaching of zinc ores or products are treated with an excess of ammonia, and carbon dioxide is passed in with agitation until the zinc is precipitated as a basic carbonate containing 3–4% of ammonia. This precipitate is granular and may readily be separated from the mother-liquor; on boiling with water it slowly loses all its ammonia and on calcination is converted into a pure white zinc oxide. A. R. POWELL.

Manufacture of aluminium chloride. G. S. TILLEY (U.S.P. 1,661,100, 28.2.28. Appl., 10.7.24).—Anhydrous aluminium sulphate is mixed with charcoal and treated at 500° with hydrogen chloride. T. S. WHEELER.

Decomposition of zirconium ores. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (B.P. 287,424, 2.11.27. Addn. to B.P. 282,023; B., 1928, 337).—The sintered mass obtained by heating zirconium ores with lime and calcium chloride or fluoride is treated with less than the theoretical quantity of hydrochloric acid required to combine with all the bases, and the concentrated solution so obtained is filtered and treated with an excess of concentrated hydrochloric acid to precipitate zirconium oxychloride. A. R. POWELL.

Manufacture of hydrogen peroxide. A. E. VIDAL. From J. D. RIEDEL A.-G. (B.P. 287,281, 4.2.27).—Hydrogen peroxide is distilled from an acid persulphate solution etc. by introducing it through a capillary tube into the lower end of a narrow vertical tube connected at the other end with a vacuum. The tube, or system of tubes, is made of chromium-nickel alloy or other chemically-resistant heavy metal, and heat is supplied internally or externally by steam, hot gases, or electrical means. W. G. CAREY.

Production of phosphorus and aluminous cement. I. G. FARBERIND. A.-G. (B.P. 287,036, 2.2.28. Ger., 19.3.27).—A mixture of bauxite, rich in iron, and lime in the proportion of 70–90 pts. of alumina to 30–10 pts. of lime is fused under reducing conditions in an electric or shaft furnace, and the easily fusible, almost iron-free slag obtained is then fused under reducing conditions with crude phosphates. W. G. CAREY.

Recovery of bromine [from saline solutions]. G. EDGAR, ASSR. to ETHYL GASOLINE CORP. (U.S.P. 1,662,305, 13.3.28. Appl., 13.10.25).—The solutions

are chlorinated and the liberated bromine is converted into an insoluble compound. F. G. CLARKE.

Isolation of argon. G. R. FONDA, Assr. to GEN. ELECTRIC Co. (1,664,205, 27.3.28. Appl., 19.3.24).—Air is partially liquefied, the liquid being less than a half of the whole, and the liquid rectified so as to obtain liquid oxygen and a distillate rich in argon. The latter is re-liquefied and rectified, in order to obtain an argon concentrate. F. G. CLARKE.

Manufacture of catalysts. H. NIELSEN, Assr. to B. LAING (U.S.P. 1,665,175, 3.4.28. Appl., 7.3.25. U.K., 3.12.21).—See B.P. 198,385; B., 1923, 760 A.

Zinc oxide [for rubber compounding]. F. G. BREYER, E. H. BUNCE, and J. H. WEIKEL, Assrs. to NEW JERSEY ZINC Co. (U.S.P. 1,664,767, 3.4.28. Appl., 18.6.25).—See B.P. 253,875; B., 1927, 107.

Separation of hafnium and zirconium. [A] D. COSTER and G. VON HEVESY, [B] A. E. VAN ARKEL and J. H. DE BOER, [C] J. H. DE BOER, Assrs. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,666,440, 1,666,800, and 1,666,811, [A—C] 17.4.28. Appl., [A] 15.7.24, [B] 11.11.24, [C] 10.8.26. [A] Denm., 18.7.23, [B] Holl., 12.12.23, [C] 12.9.25).—See B.P. 219,327, 226,180, and 258,243; B., 1925, 499; 1926, 273; 1927, 482.

Catalysts (F.P. 621,434).—See III.

VIII.—GLASS; CERAMICS.

Antimony enamels. K. BECK and W. A. SCHMIDT (Z. Unters. Lebensm., 1928, 55, 1—24).—The use of antimony compounds, especially sodium metantimonate (Leukonin), in the enamel industry is discussed. The solubility of antimony from the frit and from the enamel of cooking ware in 3% tartaric acid solution was measured, a process for the accurate determination of small quantities of mixed ter- and quinque-valent antimony being used. For enamelled ware the method is as follows:—200 c.c. of 3% tartaric acid solution are added for each 1 litre volume of the vessel, the latter is covered with a round-bottom flask filled with cold water, and the liquid boiled for $\frac{1}{2}$ hr. over a free flame with frequent shaking. After cooling, the solution is diluted to 200 c.c. To 50 c.c. are added 20 c.c. of hydrochloric acid (d 1.126) and 1 drop of methyl-orange solution (1 in 500), and the solution is titrated with 0.001N-potassium bromate solution till colourless. The titration, less a blank, multiplied by 0.0609 gives the tervalent antimony in mg. per 50 c.c. For the total antimony the solution (50 c.c.) is mixed with 20 c.c. of hydrochloric acid (d 1.126)—or the solution already titrated may be employed—and heated nearly to boiling. After removal of the flame 10 drops of 10% phosphotungstic acid are added, followed by sufficient approximately 0.1N-titanous chloride solution to give a cobalt-blue colour persisting for 2 min. After 3 min. 2 drops of 0.01% copper sulphate solution are added, and the solution should then be colourless. After a further 5 min. 1 drop of methyl-orange solution is added and the red-coloured solution titrated till colourless with 0.001N-potassium bromate. The titration, less a blank, multiplied by 0.0609 gives the total antimony in mg. per 50 c.c. J. R. NICHOLLS.

Effect of firing on the chemical and physical properties of refractories. W. MIEHR, J. KRATZERT, and H. IMMEKE (Tonind.-Ztg., 1928, 52, 280—282, 298—301, 323—325).—The relation between chemical composition of refractories and mullite formation at or above 1000° is discussed briefly. The mullite formation between 1000° and 1600° was determined by chemical means in 12 clays of different chemical and mineralogical composition, and the relation between mullite content and the temperature and duration of firing, grain size of the material, clay-substance content, and alkaline flux content was studied. The results were confirmed and extended by microscopical and X-ray investigations. Mullite is formed most readily, i.e., at relatively low temperatures, in clays rich in fluxes and poor in clay substance; in this case its formation can be facilitated by fine grinding of the material. A long firing period at a very high temperature is necessary to produce mullite in clays rich in clay substance and poor in fluxes; an increase in the flux content of such clays lowers the temperature of mullite formation somewhat. The effect of mullite formation and the accompanying structural changes on the crushing strength, thermal expansion (resistance to spalling), and the resistance to chemical action (slags) of refractories is discussed. F. SALT.

Properties of magnesite bricks. H. HIRSCH (Arch. Eisenhüttenw., 1927, 1, 439—444; Stahl u. Eisen, 1928, 48, 371—372).—The physical, mechanical, and refractory properties of magnesite bricks from 32 different sources have been determined, and from the results obtained the samples have been divided into three groups, viz., those containing more than 7%, those with 3—7%, and those with less than 3% Fe_2O_3 . These groups may again be subdivided into nine sub-groups the characteristics of which are tabulated. Magnesite bricks containing only silica as an impurity have a softening point under load of about 1790°, which approaches that of bricks made of sintered magnesite; they have a high true sp. gr. and low porosity, but a relatively high coefficient of expansion. Although the behaviour of the bricks under load varied greatly with the composition, the refractoriness varied only slightly, all the samples withstanding exposure to a temperature above cone 42. X-Ray examination of well-burnt bricks showed that practically the whole of the magnesite has been converted into periclase, and therefore no further transformation should take place even after prolonged heating. Bricks containing 5—7% Fe_2O_3 and very small amounts of silica have the best sintering properties and the greatest mechanical strength.

A. R. POWELL.

Composition of "Kochi-stone." FUKUCHI and WATANABE (J. Geol. Soc., 1925, 32, 131—141).—This refractory material contains SiO_2 24.68, Al_2O_3 61.80, Fe_2O_3 0.60, CaO 0.60, MgO 0.10, F 5.18, Cl 2.91, H_2O 6.15%.

CHEMICAL ABSTRACTS.

PATENTS.

Kilns for firing pottery, tiles, etc. T. SIMPSON (B.P. 285,935, 21.10.26).—The fire mouths of the kiln extending underneath the kiln floor are each divided by a short central midfeather, and have a set of up-

flues at the front and back to convey the heat from each mouth to the kiln crown. Passages in the midfeathers direct the heat along the crown to the back down-flues, which convey the heat underneath the kiln, whilst up-flues at the front of the kiln convey the heat to a conduit leading to a chimney. A damper on the outside wall of the kiln facilitates cooling and allows the kiln to be drawn in a shorter time than usual. W. G. CAREY.

Apparatus [tunnel kiln] for firing ceramic and other products. C. T. HOLCROFT and C. E. DOLL (U.S.P. 1,662,063, 13.3.28. Appl., 1.12.24).—In a tunnel kiln of the muffle type the air is circulated in direct contact with the articles as they advance through the kiln. The air supply, which is under pressure, is near one end of the tunnel. F. G. CLARKE.

Manufacture of appliances and articles of highly refractory material such as oxide of zirconium, of thorium, and of the rare earths. DEUTS. GAS-GLÜHLICHT-AUER-GES.M.B.H. (B.P. 280,907, 12.11.27. Ger., 22.11.26).—Articles of zirconium oxide, thorium oxide, etc. are improved in solidity and density if the mixture of sintered and unsintered material is heated as rapidly as possible to 1300° or above. W. G. CAREY.

Crucibles [for molten metals]. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of M. UNGER (B.P. 278,367, 28.9.27. U.S., 1.10.26).—Leakage of molten metal through porous fireclay crucibles is prevented by impregnating them, preferably *in vacuo*, with a liquid, e.g., coal-tar pitch, containing a refractory substance in suspension or solution, and slowly heating them to drive off the volatile components and leave carbon in the pores of the crucible. W. G. CAREY.

Manufacture of chamotte materials. L. KERN (U.S.P. 1,664,689, 3.4.28. Appl., 13.10.25).—A grog material suitable for use in chamotte is produced by burning at a strong glowing heat a plastic mass of kaolin, combustible organic matter, and a binder, all in a finely-divided state, and then pulverising the product. H. ROYAL-DAWSON.

Compositions for heat-insulating and heat-resisting purposes. L. F. TOOTH (B.P. 287,363, 22.6.27).—The composition, which can be shaped into bricks or used *in situ*, comprises Portland cement or fireclay, slagwool, and crushed burnt firebrick (grog), e.g., in equal pts. by wt., with the addition of 0.33–1.0% of aluminium powder or similar material, which on heating causes the evolution of gas and the formation of a porous product. L. A. COLES.

Manufacture of pottery etc. J. W. MELLOR (U.S.P. 1,666,828, 17.4.28. Appl., 14.1.26. U.K., 9.3.25).—See B.P. 253,184; B., 1926, 667.

Glass furnaces. HARTFORD-EMPIRE CO., Assees. of P. G. WILLETTTS (B.P. 275,555, 22.3.27. U.S., 3.8.26).

Manufacture of imitations of frosted glass, glass stainings, etc. R. GOLDSCHNEIDER and I. TUSCHINSKY (B.P. 286,012, 29.11.26).

Ovens of the direct flame continuous tunnel type. J. WILLIAMSON (B.P. 287,623, 22.12.26).

Treatment of sludge (B.P. 286,855).—See I. **Magnesia refractory** (U.S.P. 1,664,631).—See VII.

IX.—BUILDING MATERIALS.

Electrical method for measuring the setting time of Portland cement. Y. SHIMIZU (Sci. Rep. Tōhoku Imp. Univ., 1928, 17, 85–92).—On measuring the electrical conductivity of gauged cement during setting and hardening, the specimen being maintained at constant temperature, the resistance suddenly changed at the moment corresponding to the final set, as defined by the maximum temperature change. The change in resistance is used to determine the time of setting, the mixed cement being contained in a small tube in a thermostat; two platinum electrodes are immersed in the cement and connected to a Kohlrausch alternating-current bridge. The rate of setting and hardening is influenced greatly by temperature, but the method is considered better adapted and more accurate than the mechanical and thermal methods. C. A. KING.

Formation and dissolution of calcium hydroxide crystals in Portland cement. J. O. DRAFFIN (Ind. Chem., 1928, 20, 311).—After the setting of Portland cement, crystals of calcium hydroxide gradually form, originating from the hydrolysis of tricalcium silicate. In a study of this process on microscope slides such crystals later were found to redissolve, apparently owing to slight dissolution of the glass of the slide. No such dissolution could be produced with natural soda-lime felspar. C. IRWIN.

Mechanism of the action of wood-preserving agents. DEHNST (Z. angew. Chem., 1928, 41, 355–358).—To test Bateman's theory (Proc. Amer. Wood Preservers' Assoc., 1920–1924) that the fungicidal action of coal-tar oil or creosote oil is due to the low-boiling hydrocarbons and the high-boiling tar acids and bases and is not influenced in any way by the 40% of neutral oil usually present, a very pure German coal-tar oil was freed from acids and bases and separated into several fractions by distillation, and the action of the various products on four of the ordinary varieties of wood fungus was examined. All the products obtained had a strong fungicidal action on *Polyporus vaporarius* and *Coniophora cerebella*, but the neutral oil boiling above 288° had little action on *Lenzites abietina* and *Fomes annosus*, although the remainder of the oil had a strong fungicidal action on these varieties. A. R. POWELL.

PATENTS.

Burning of cement. A. ANDREAS (B.P. 263,166, 15.12.26. Ger., 16.12.25).—Air is blown at a relatively high pressure into a kiln for burning cement at a point directly below the clinkering zone and also, at the same pressure, at lower points, including one below the grinding rollers, if desired. The air is introduced through pipes surrounding the kiln at suitable heights, fitted with branch pipes at regular intervals leading to the interior of the kiln. The kiln tapers towards the grinding rollers or outlet at the lower end. L. A. COLES.

Brick making. G. H. HADFIELD (B.P. 286,772, 7.12.26).—Clinker relatively free from carbonaceous material recovered from boiler ash, house refuse, etc. is used as opener-out in the manufacture of bricks from plastic clay. L. A. COLES.

Manufacture of acid-proof cementing compositions. I. G. FARBERIND. A.-G. (B.P. 281,689, 1.12.27. Ger., 1.12.26. Addn. to B.P. 258,616; B., 1927, 333).—In place of silicon an alloy of silicon with, *e.g.*, magnesium, aluminium, or calcium, which consists partly of pure silicon or of silicon containing other constituents in solid solution, is used. L. A. COLES.

Filling cracks in the brickwork of ovens. G. CAPIAU, M. GAUQUIER, and L. LAHAUT (B.P. 286,933, 7.6.27).—A dry, finely-powdered mixture of refractory cement, a flux (*e.g.*, dextrin or borax), litharge, sodium chloride, and sodium carbonate is forced into cracks in the brickwork of coke ovens etc. while the brickwork is still hot. L. A. COLES.

Manufacture of asphaltic, bituminous, or tarry emulsions. SOC. ANON. "LA TRINIDAD" (B.P. 264,496, 11.1.27. Fr., 12.1.26).—An anhydrous mastic prepared by adding a mineral powder, *e.g.*, cement or powdered limestone, to products such as asphalt, bitumen, or pitch, is emulsified by mixing with a suitable agent such as soap. The product is suitable for the construction of roads. A. B. MANNING.

Preservation of timber. H. C. GARDINER (B.P. 286,886, 19.3.27).—The portion of wooden posts etc. buried in the ground is protected against rot by placing in contact with it a slightly soluble powdered preservative containing arsenic, *e.g.*, arsenic trioxide or arseniferous smelting residues, and surrounding it with a pervious covering so that the slow percolation of water causes continuous impregnation of the wood and sterilises the surrounding soil. Deliquescent substances, *e.g.*, sodium chloride, magnesium chloride, etc., or compounds capable of increasing the solubility of the material, *e.g.*, sodium carbonate, may be added to the preservative. L. A. COLES.

Preservation of wood, timber, etc. G. GUNN (B.P. 286,892, 25.3.27. Addn. to B.P. 273,007; B., 1927, 655).—The timber, treated as described in the prior patent, is drained and exposed to the action of steam. L. A. COLES.

Manufacture of burnt building materials etc. of clay. H. GROŇROOS (U.S.P. 1,664,815, 3.4.28. Appl., 4.8.25. Denm., 6.8.24).—See B.P. 238,229; B., 1926, 632.

Production of artificial stone and artificial wood. F. TRACHSEL, J. WIEDNER, and P. ZIGERLI (U.S.P. 1,665,148, 3.4.28. Appl., 3.5.26. Switz., 1.5.26).—See B.P. 280,104; B., 1928, 93.

Making of road surfaces. M. LÉVY, and PRODORITE S.A. (B.P. 273,759, 2.7.27. Ger., 2.7.26).

[Emulsification] apparatus for dealing with bitumen for use as a road material etc. H. E. WARSOP (B.P. 287,257, 1.1.27).

Aluminous cement (B.P. 287,036).—See VII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Magnetic concentration of iron ores of Alabama. High-silica red ores. High-silica grey hæmatite of Talladega county. Flue dust of the Birmingham

district. O. LEE, B. W. GANDRUD, and F. D. DE VANEY (U.S. Bur. Mines, Bull. 278, 1927, 1—33, 35—56, 61—75).—Results with high-silica red ores closely approached theoretical limits. Flow-sheets are given. Roasting in a reducing atmosphere at 500—600° converts the hæmatite into magnetite, often eliminating the necessity for fine grinding. The ores are mostly oolitic hæmatites. Treatment of the grey ore, a mixture of hæmatite and magnetite, is described; 94—95% of the iron is extracted. The treatment of flue dust is described. CHEMICAL ABSTRACTS.

Magnetic concentration of iron ores of Alabama. Flotation of limestone from siliceous gangue. O. LEE (U.S. Bur. Mines, Bull. 278, 1927, 57—59).—The results are tabulated; equal quantities of oleic acid and cresol are used as flotation agents. CHEMICAL ABSTRACTS.

Influence of occluded oxygen in steel on the carburising quality of steel. K. INOUE (Mem. Coll. Eng. Kyushu Imp. Univ., 1928, 5, [1], 69 pp.).—Samples of mild steel, prepared by melting the metal *in vacuo* and giving products containing 0.04—0.23% O as determined by Oberhoffer's method, were carburised. The critical points of steel were not affected by change in the oxygen content, but the specific resistance increased with increasing oxygen, though not following Mahler's formula. Grain growth was noted in steels containing 0.116% O or more. The carburising experiments were made to test the view that a larger grained structure facilitates the penetration of carbon; the results supported the theory and failed to confirm the view that the presence of oxygen generally induces shallow case-hardening. C. A. KING.

Equilibria in the reduction, oxidation, and carburisation of iron. R. SCHENCK (Stahl u. Eisen, 1928, 48, 269—271, 274—276). E. MAURER and W. BISCHOF (*ibid.*, 271—274, 276).—Polemical. Schenck replies to criticisms of his work (A., 1927, 939, 1030) by Maurer and Bischof (Stahl u. Eisen, 1928, 48, 15) and gives fuller experimental details. The latter authors maintain and amplify their previous criticisms. A. R. POWELL.

Duralumin change in iron-carbon alloys. G. MASING and L. KOCH (Wiss. Veröff. Siemens-Konz., 1927, 6, 202—210).—A change similar to that which occurs with duralumin and other aluminium alloys after rapid cooling from about 500° and subsequent keeping at ordinary temperature or heating again to about 150°, occurs also with iron-carbon alloys. The change which occurs is distinguished from the result of ordinary quenching, and is a modification with time of the properties of the alloy, particularly of the hardness. The observations were made on various irons which were first heated to 660—700°, quenched, and then kept for several weeks, the hardness being measured at intervals; it was found that an increase, amounting to about 80% of the original hardness, took place during two to three weeks. S. I. LEVY.

Theory of steel hardening. K. HONDA (Arch. Eisenhüttenw., 1927—8, 1, 527—536; Stahl u. Eisen, 1928, 48, 263—263).—The changes taking place when steel is cooled from above the A1 point to 20° may be summarised as follows: austenite (tetragonal lattice *c/a* =

1.414)→ α -martensite (tetragonal lattice $c/a = 1.04$) → β -martensite (cubic body-centred lattice)→ pearlite (precipitation of carbon). With very slow cooling these changes proceed to completion; with slightly more rapid cooling part of the martensite remains undecomposed and the steel is partly hardened; with a still greater rate of cooling the change to pearlite is suppressed, the structure consists of ($\alpha + \beta$)-martensite, and the steel is completely hardened; and, finally, with a greater rate of cooling, austenite remains undecomposed on the outside of the specimen and the steel is then overhardened. Annealing of hardened steel at 150° causes the α -martensite to be converted into the β -modification with an increase in the intensity of magnetisation, a decrease in the electrical resistance, and a decrease in volume; at 250° β -martensite is converted into pearlite with evolution of heat but without change of volume. At a somewhat higher temperature a considerable contraction in volume takes place accompanied by a change in the magnetic properties and by absorption of heat. As cementite is the only endothermic phase that can exist at this temperature, these facts can be explained only on the assumption that the decomposition of the solid solution (β -martensite) takes place in two stages, in the first of which carbon atoms are displaced from the lattice and in the second of which these atoms combine with iron to form the carbide, cementite, with absorption of heat and a decrease in volume. The greater hardness of martensite compared with ferrite, although both have a similar lattice structure, is ascribed to the finer grain of martensite, the presence of internal stress due to a state of metastable equilibrium, and the presence of carbon atoms in the lattice in such a way that the lines of force emanating from them affect six neighbouring iron atoms. Assuming that ferrite has a Brinell hardness of 225 the increase due to the above-named factors would be 80, 150, and 225 respectively, giving the martensitic hardened steel a Brinell hardness of 680. A. R. POWELL.

Quenching of steels in a high-temperature bath. K. HONDA and K. TAMARU (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 69—83).—Quenching of steels in low-temperature media, *e.g.*, water and oil, is often accompanied by cracking, visible or microscopic, in either case harmful, and according to the theory of quenching advanced by Honda (B., 1919, 821 A) there is no reason why troostitic and sorbitic structures should not be obtained by a mild quenching in a hot bath. Austenite is transformed into pearlite through martensite irrespective of the cooling velocity, and when this change occurs at a high temperature the martensite is converted again into troostite or sorbite. Since the change austenite → martensite → troostite involves an expansion and a contraction, the two phases altering side by side causes no abrupt change in volume. A mixture of 65% of potassium nitrate and 35% of lithium nitrate (m.p. 132°) was used as a quenching medium above 150°, for lower temperatures mercury was utilised, when quenching a steel containing 0.89% C. Maximum hardness was obtained at about 110°, and impact-resistance increased gradually up to 320°, decreasing at higher temperatures. Specimens quenched in the salt bath below 530° were weaker than under normal treatment,

but had a higher tensile strength when quenched at above 530°. Quenching cracks occurred always at temperatures below 120°, but any desired structure could be obtained without any risk of cracks if the bath was above 150°, martensitic up to 200°, troostitic 300—400°, and sorbitic above 400°. C. A. KING.

Diffusion phenomena of zinc and tin in steel and soft iron. H. HONIGL (Mikrochem., 1928, 6, 22—27).—Tin begins to diffuse into iron and steel at 300° with the formation of a thin layer of solid solution which at this temperature appears to hinder further diffusion. At 500—750°, however, diffusion of tin into steel proceeds rapidly with the formation of several layers of inter-metallic compounds interspersed with crystals of solid solutions; at the same time the carbon content of the steel diffuses towards the central portions, leaving a layer of decarburised steel inside the layers of tin-iron compounds. At 900° there is a pronounced tendency for the carbon to diffuse back towards the edges of the test-piece. The rate of diffusion of zinc into iron increases rapidly with the temperature above 500° with the formation of three distinct layers of differently oriented mixed crystals, accompanied by an enrichment of the carbon content of the inside portions of the test-piece. A. R. POWELL.

Effect of a short period of annealing on the tensile strength and electrical conductivity of copper, bronze, aluminium, aludur, and aldreyl. H. BOHNER (Z. Metallk., 1928, 20, 132—141).—Experiments on hard-drawn wires (99% reduction) of copper (99.95% Cu), bronze (containing small quantities of magnesium and tin in solid solution), aluminium (containing 0.17% Fe and 0.14% Si), aludur (aluminium containing 0.27% Fe, 1.07% Si, and 0.43% Mg), and aldreyl (aluminium containing 0.33% Fe, 0.49% Si, and 0.51% Mg) are described in which the electrical conductivity and tensile strength after passing a heating current through them for varying short periods of time were determined. For metals and alloys consisting wholly of solid solution at the ordinary temperature complete recrystallisation, accompanied by minimum tensile strength and maximum electrical conductivity, is obtained in a fraction of a second at a sufficiently high temperature, *e.g.*, 700° for copper, 550° for the above bronze. This also applies to alloys which require to be aged at a high temperature to obtain the maximum tensile strength, *e.g.*, aldreyl. On the other hand, alloys of which the strength and conductivity depend on the form in which the constituents are present require a relatively long period of annealing when the annealing temperature necessary to produce minimum strength and maximum conductivity is low; this is due to the slow rate of diffusion of the constituents. A. R. POWELL.

Influence of dissolved gases on the soundness of 70:30 brass ingots. G. L. BAILEY (Inst. Metals, March, 1928. Advance copy, 12 pp.).—Ingots cast from brass treated in the molten state with nitrogen, hydrogen, and sulphur dioxide were cast in a series of moulds giving a wide range of cooling velocities. In no case was evidence obtained of unsoundness caused by gas evolution, though slow speeds of cooling were associated with extensive shrinkage cavities. It is considered that the high vapour pressure of zinc in molten brass

precludes the dissolution of gases, and that the spherical cavities occurring in brass ingots are the result of gases trapped mechanically within the mould. The conclusion is opposed to that of Bamford and Ballard (B., 1921, 84 A) as brass can be cast safely by special methods from only 30° above the liquidus. C. A. KING.

Quenching and tempering of brass, bronze, and "aluminium bronze." T. MATSUDA (Inst. Metals, March, 1928. Advance copy. 42 pp.).—When quenched from high temperatures the hardness of ($\alpha + \beta$)-brass increased while the electrical resistance decreased, but no abnormal change corresponding to the β -transformation was observed. No appreciable change in the properties of a bronze containing 10–12% Al occurred if quenched below 600°, but the hardness and electrical resistance increased steadily due to redissolution of α in β . A bronze containing 18–20% Sn behaved similarly to the aluminium bronze, showing, however, another decrease in hardness at the eutectoid transformation at 590°; in all cases the rate of cooling had a pronounced effect. The temper-hardening is due probably to the straining of space-lattice produced by the separation of α from β or γ , or the decomposition of β or γ into eutectoid. C. A. KING.

Determination of antimony in bronze, brass, and similar alloys. S. A. TSCHERNICHOV (Z. anal. Chem., 1928, 73, 265–270).—The alloy (5 g.) is dissolved in nitric acid and the precipitate of metastannic and antimonious acids is collected, ignited, and dissolved by heating with concentrated sulphuric acid and a few crystals of sugar. When the acid becomes colourless it is cooled and diluted with water, hydrochloric acid is added, and the antimony titrated as usual with bromate. If the alloy contains no tin the addition of a small quantity of this metal to the flask in which the dissolution is effected is necessary in order to ensure that the antimony is completely precipitated.

A. R. POWELL.

Thermal and electrical conductivity of some aluminium alloys and bronzes. E. GRIFFITHS and F. H. SCHOFIELD (Inst. Metals, March, 1928. Advance copy. 36 pp.).—A series of seven copper alloys containing 0.5–12.4% Sn and 2% Zn showed thermal conductivities at room temperature varying from one fifth to one tenth of that of copper, increasing considerably with rise of temperature in contrast to the conductivities of pure metals. An admixture of 10% of aluminium was comparable in its effect with that due to 10% of tin, and minute traces of phosphorus gave rise to appreciable reduction in conductivity. All the alloys followed Lorenz's law of the relation between thermal and electrical conductivity and absolute temperature. In the series of light aluminium alloys, the two containing 4.5% and 8% Cu, respectively, possessed a thermal conductivity equal to 82% of that of aluminium. Alloys containing copper, nickel, iron, and manganese in increasing quantities gave decreasing conductivity values, the lowest value of the series being given by an alloy containing 13% Zn and 3% Cu. The Lorenz coefficients of aluminium alloys were consistent but of lower value than those for copper alloys. C. A. KING.

Recrystallisation of the silver[–copper] alloy containing 80% Ag. R. GLOCKER and H. WIDMANN

(Z. Metallk., 1928, 20, 129–131).—The tensile strength of the 20:80 copper–silver alloy after cold-rolling to 98% reduction is about 90 kg./mm.² compared with 40 and 50 kg./mm.² for similarly treated silver and copper respectively. The tensile strength of the worked alloy decreases slowly with rise in annealing temperature to 85 kg./mm.² at 200°, then more rapidly and almost linearly to 35 kg./mm.² at 600°, and, finally, slowly again to 30 kg./mm.² at 800°. The tensile strength of pure silver, on the other hand, decreases suddenly at 200° to a constant value of 30 kg./mm.² Annealing the alloy at 800° produces new grains which in 30 min. are 2–5 times as great as those produced in 13 hrs. at 780°. Microscopical examination with a high-power objective fails to disclose any sign of recrystallisation below 500°, and X-ray examination by the Debye-Scherrer method shows that the characteristic rolled structure persists up to 780°, and that recrystallisation is complete only after 1 hr. at 800°. The broadening of the outer rings in the X-ray photographs, however, begins to disappear on annealing at 300°, and at 400° these rings become quite sharp, showing that distortion of the lattice caused by the cold-work has been relieved. Hence this method affords a delicate test for determining the temperature at which the preliminary rearrangement of the atoms prior to recrystallisation takes place. A. R. POWELL.

Dilatometric study of light metals. M. HAAS (Inst. Metals, March, 1928. Advance copy. 16 pp.). Technical improvements in the working of the Oberhoffer-Esser dilatometer are described. The chief heating tube of the furnace consists of nickel with a network of chrome-nickel heating strip. Copper heads at the end of the tube serve as heat reservoirs, and the whole is surrounded by a second nickel tube wound for uniform heating. The use of a test bar having rounded ends and also of delicate pressure contact springs is recommended to minimise the increasing plasticity of a test-piece at higher temperatures. Curves of several light alloys are reproduced. C. A. KING.

Tin bronzes. M. HANSEN (Z. anorg. Chem., 1928, 170, 18–24).—Bands round the edges of the crystal grains may appear after etching in alloys of copper with 15–25% of tin when quenched even from below 590°. The formation of the bands is, therefore, not connected with the transition point at 590°, and is not a consequence of a polymorphic transformation of the β -crystals, as Stockdale has suggested (B., 1925, 809). The bands are the wider the slower is the quenching, and the nearer the temperature from which the alloy is quenched approaches to 520°. It thus appears likely that they consist of a crystal species stable at lower temperatures, and formed in spite of the quenching. This could only be the δ -crystal, formed at 520°, but this cannot be the only constituent of the bands, since crystals and bands may be present together. It seems, therefore, highly probable that it is the ($\alpha + \delta$)-eutectoid in a very highly dispersed state which is involved, and it has in fact been possible to reproduce various intermediate stages between the bands, apparently consisting of only one phase, and the visibly two-phase eutectoid. R. CUTHILL.

Volume change in certain type metals during solidification. Y. MATUYAMA (Sci. Rep. Tôhoku Imp.

Univ., 1928, 17, 1—25).—The change in volume of certain metals and alloys used in casting type has been measured by means of a thermo-balance. Pure antimony showed a volume expansion during solidification of 0.95% (approximately one third that of bismuth), but a contraction was observed in every trial with standard type alloys. As this is contrary to general belief it is pointed out that the surface tensions of antimony and lead, which are the chief components of type metals, are smaller than those of many other pure metals, and so a good fluidity of the melt is produced. Also it is usual to apply pressure on the molten metal in the type mould which aids in securing "sharp" type. C. A. KING.

Reversed segregation [in mixed-crystal alloys]. II. G. MASING and C. HAASE (Wiss. Veröff. Siemens-Konz., 1927, 6, 211—221; cf. Bauer and Arndt, B., 1922, 220 A).—The various explanations of the fact that the first-cooled portions of a casting of an alloy of mixed crystal structure are found to be richer than the remainder in the most fusible component are considered.

S. I. LEVY.

[Standard specifications for] metals. AMER. SOC. TESTING MATERIALS (Part I, Metals, 1927, 871 pp.).—Standard specifications, methods of testing, definitions, and rules are given, together with certain recommended practices.

CHEMICAL ABSTRACTS.

PATENTS.

Carrying-out of the Bessemer process [for steel]. A. E. WHITE. From S. G. ALLEN (B.P. 286,745, 30.8.26).—The "net blow-heat," i.e., the gross heat developed by oxidation in the molten charge by the oxygen in the blast less the quantity of heat absorbed in raising the temperature of the waste gases, of any particular charge is determined and the proportion of oxygen in the blast is regulated to minimise loss of heat in the waste gases. [Stat. ref. to B.P. 20,468 of 1904 and 13,952 of 1900.] C. A. KING.

Furnace for treating iron with a low proportion of carbon. F. WÜST (B.P. 275,646, 5.8.27. Ger., 6.8.26).—Scrap iron or steel is melted in a cupola furnace connected to a continuously working reverberatory furnace. A substantial proportion of the heat required for melting is supplied from the reverberatory furnace and the coke charge to the cupola is regulated within the carbon limit of the iron, which is superheated in the second furnace to ensure the formation of eutectic graphite. C. A. KING.

Apparatus for purifying and de-aerating molten iron. J. DECHESNE (B.P. 271,100, 13.5.27. Ger., 14.5.26. Addn. to B.P. 265,827; B., 1927, 302).—During the pouring of molten iron from a furnace into the ladle the latter is mechanically agitated; e.g., the ladle may be tiltably journalled and jiggled by means of cams underneath. C. A. KING.

Production of synthetic pig iron. A./S. NORSK STAAL (ELEKTRISK-GAS-REDUKTION), and E. EDWIN (B.P. 286,067, 12.2.27).—Spongy iron, produced by reducing iron ore and separating the gangue before melting, is melted with substantially pure carbonaceous material, together with additions of silicon and man-

ganese, in an electric furnace at 1100—1300° under acid conditions. C. A. KING.

Manufacture of commercially pure iron alloys. L. F. REINARTZ and J. H. NEAD, Assrs. to AMER. ROLLING MILL Co. (U.S.P. 1,665,146, 3.4.28. Appl., 4.10.23).—A bath of molten iron low in carbon and metalloids is treated in an electric furnace with a degasifying agent, then with a reducing slag, and finally with an alloying element substantially free from carbon. A. R. POWELL.

Protection of ferrous metals against corrosion. G. B. ELLIS. From T. E. MURRAY (B.P. 287,194, 8.11.26).—Iron and steel are electroplated with nickel on which a layer of chromium is subsequently applied. The product is then heated at 980—1380° in an oxidising atmosphere. Partial alloying occurs and the outer surface is covered with a thin tenacious film of oxide. C. A. KING.

Galvanising or coating by dipping of steel. E. A. ATKINS, and RYLANDS BROS., LTD. (B.P. 287,201, 19.11.26).—In a galvanising process, steel articles are immersed in lead or a lead alloy maintained at 600—800° and are then immersed in molten zinc without coming into contact with the atmosphere. In practice, a layer of molten zinc may float on the surface of the lead bath. C. A. KING.

Manufacture of chromium or similar refractory metals. GEN. ELECTRIC Co., LTD., and C. J. SMITHELLS (B.P. 285,571, 18.11.26).—Coherent articles of chromium are formed by agglomerating the pure powdered metal into shapes and sintering it at 1500—1550° in hydrogen freed from oxidising impurities. Hydrogen for this purpose may be prepared by passing the gas through a vessel containing incandescent tungsten and then through a tube cooled to the temperature of liquid oxygen. C. A. KING.

Mechanical ore-roasting furnace. J. HARRIS (B.P. 287,356, 2.6.27).—An upper hearth of a multiple-hearth furnace is provided at its discharge end with a conduit depending from the hearth above the rabble arm of the hearth below. Ore falling into the conduit may be retained there by a sealing-plate carried by the rabble shaft until, by the rotary motion, registration is effected with a second conduit which allows the ore to be guided to the lower hearth out of contact with the furnace draught and so without the production and loss of dust. C. A. KING.

Melting of metals. F. W. CORSALLI (B.P. 267,972, 19.3.27. Ger., 19.3.26).—Refining metals for addition to the charge of a cupola or shaft furnace are melted separately in a furnace adjoining, and are introduced directly into the melting zone of the cupola. C. A. KING.

Ore- or like mixing machines. H. ERIKSSON (B.P. 287,016, 2.11.27).—A machine for mixing ore concentrate with coal or coke breeze consists of a vertical, open-ended, rotary drum supported above a stationary bottom plate having an adjustable aperture for the discharge of materials. The drum is provided with a series of cross-bars which are spaced in relation to beaters on a central shaft. C. A. KING.

Sifting or screening apparatus for the classification of ores etc. A. FRANCE (B.P. 276,378, 17.1.27. Belg., 18.1.26).—A screening channel having an increasing inclination towards the discharge end is so mounted that a rotary movement is imparted to the receiving end, *e.g.*, by mounting it on a crank of a driving shaft, and a vertically-oscillating movement is given to the discharge end.

C. A. KING.

Flotation processes. D., M., S. R., and S. GUGGENHEIM, J. K. MACGOWAN, and E. A. C. SMITH (GUGGENHEIM BROS.), Assees. of A. H. FISCHER (B.P. 284,198, 29.4.27. U.S., 24.1.27).—Flotation agents comprising the products obtained by the interaction of a metal xanthate or metal alkylxanthate with an acid chloride, such as carbonyl, acetyl, or sulphuryl chloride, or with an alkyl chloroformate are claimed. This class of flotation agents is particularly suitable for working in acid media.

A. R. POWELL.

Extraction of metallic values [copper, lead, and zinc] from ores. F. DIETZSCH (B.P. 287,207, 8.12.26 and 16.2.27).—Oxidised or roasted copper ores are leached with a cold solution of sodium or calcium thiosulphate saturated with sulphur dioxide, whereby the cuprous sulphite first formed is dissolved by the thiosulphate solution. Addition of lime to the leach liquors precipitates copper hydroxide with the regeneration of the thiosulphate solution. For the treatment of ores containing lead sulphate the use of sulphur dioxide is unnecessary, otherwise the same process is applicable to oxidised lead ores and zinc ores. Oxidised lead-zinc ores are first leached with sulphur dioxide solution alone to dissolve the zinc and the residue is then leached with thiosulphate solution.

A. R. POWELL.

Reduction of ores. H. WITTEK (B.P. 275,144, 20.11.26. Ger., 27.7.26. Addn. to B.P. 274,803; B., 1928, 269).—Ores are reduced in combination with the nitrogenation of carbides of metals other than calcium, the carbides of which are capable of nitrogenation.

C. A. KING.

Manufacture of tinned plates. H. WILLIAMS (B.P. 287,288, 9.2.27).—Plates proceeding from a tinning bath are passed through water at 90–100° so that the plates leave the water-bath hot and any water adhering to the plates is evaporated rapidly.

C. A. KING.

Application of thin, reflecting, metal coatings to the surface of flexible or elastic materials. LAMPEN- U. METALLWAREN-FABR. R. DITMAR GEBR. BRÜNNER A.-G., Assees. of A. DOMAN (B.P. 262,827, 14.12.26. Hung., 14.12.25).—The material to be coated is impregnated with a binding material, *e.g.*, resin or gum, laid on the thin reflecting film of metal, and gently heated to cause the two surfaces to adhere. Alternatively, a layer of an emulsion or binding material is applied to the back of the metal film and the fabric is pressed on to this layer before it dries completely. The metal film is deposited on to a reflecting surface by a galvanoplastic process and peeled therefrom after fixing the elastic material to its upper side.

A. R. POWELL.

Joining together of metal surfaces. W. SMITH (B.P. 285,995, 25.1.26 and 1.9.27).—Articles of similar or dissimilar metals are united by pouring molten metal to come into contact with the amalgamated surfaces of the

articles; *e.g.*, articles of aluminium are joined by placing a strip of amalgamated zinc against each surface to be joined and pouring molten aluminium between the strips.

C. A. KING.

Recovery of light metals from scrap. I. G. FARBERIND, A.-G., and A. BECK (B.P. 287,360, 13.6.27. Addn. to B.P. 182,948; B., 1922, 715 A).—Anhydrous calcium chloride containing additions of calcium fluoride may be used as a flux, the eutectic composition of the melt (82% CaCl₂, 18% CaF₂) being avoided.

C. A. KING.

Alloys for bearings. W. MATHESIUS and M. NEUFELD (F.P. 622,237, 28.9.26. Ger., 1.10.25).—Further to B.P. 259,211, alloys containing 0.2–0.25% Mg together with 0.1% Bi, Cu, Sn, or of mixtures of two or all of these metals are claimed.

C. HOLLINS.

Non-ferrous casting [alloy]. R. L. BINNEY, Assr. to BINNEY CASTINGS Co. (U.S.P. 1,665,435, 10.4.28. Appl., 6.7.25).—A refractory alloy containing, approximately, 1 pt. of manganese, 2 pts. of zinc, 18 pts. of copper, and $\frac{1}{2}$ pt. of aluminium is claimed.

F. G. CROSSE.

High-resistance alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 285,565, 17.11.26).—The alloys, resistant also to oxidation, contain 70–80% (75%) Ni, 15–25% (20%) Cr, 2–7% (5%) Co. Iron may be present up to 10%, about half of the iron replacing chromium in the alloy.

C. A. KING.

[Non-magnetic] alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 287,236, 17.12.26).—The alloy of high electrical resistance contains 23–27 (23.7%) Ni, 71–67 (70.8%) Fe, 1.5–4 (3%) Cr, and 1.5–4 (2.5%) Al or Si.

C. A. KING.

Enamelled wires. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 274,079, 4.7.27. Holl., 12.7.26).—Wires, resistant to oxidation and suitable for wireless transformers, have a core of a highly refractory metal, *e.g.*, molybdenum or alloy.

C. A. KING.

Thermostatic material. P. H. BRACE, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,660,911, 28.2.28. Appl., 12.3.25).—A thermostatic strip comprises elements of molybdenum and of an alloy of electrolytic iron and chromium.

T. S. WHEELER.

Briquetting [of iron ores etc.]. A. CARLSON (U.S.P. 1,666,618, 17.4.28. Appl., 8.4.25. Swed., 10.5.24).—See B.P. 233,731; B., 1925, 962.

Production of rustproofing material [for iron]. H. H. WILLARD and M. GREEN, Assrs. to PARKER RUST-PROOF Co. (U.S.P. 1,660,661, 28.2.28. Appl., 10.5.26).—See B.P. 273,168; B., 1927, 658.

Volatilisation and oxidation of minerals. B. G. COBB, Assr. to COBB ELECTRO REDUCTION CORP. OF CANADA, LTD. (U.S.P. 1,660,150, 21.2.28. Appl., 6.4.21. Renewed 1.3.27).—See B.P. 223,271; B., 1925, 13.

Apparatus for the manufacture of tin plates. G. B. ELLIS. From AMER. SHEET & TIN PLATE Co. (B.P. 287,436–7, 13.9.26).

Crucibles (B.P. 278,367).—See VIII. **Emulsions for tin-plating machines** (B.P. 287,438).—See XII.

XI.—ELECTROTECHNICS.

Setting time of cement. SHIMIZU.—See IX. **Concentration of ores.** LEE and others. **Conductivity of metals and alloys.** BOHNER, also GRIFFITHS and SCHOFIELD.—See X.

PATENTS.

Electric furnace. J. L. MCFARLAND, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,661,842, 6.3.28. Appl., 16.6.27).—Insulating supports, from which a ribbon-shaped heating resistor is hung in the form of a series of depending hoops, are secured to the refractory walls of the heating chamber. Headed spacing members are secured to the furnace wall in alternate spaces between the lower ends of the loops, the heads overhanging the lengths of the adjacent resistor. Parallel-sided, wedge-shaped spacing members for the loops project from the wall in the remaining spaces between the lower ends of the loops, the edges of which members extend horizontally.

J. S. G. THOMAS.

Electric [induction] furnace. C. B. FOLEY, Assr. to C. B. FOLEY, INC. (U.S.P. 1,661,173, 6.3.28. Appl., 23.3.22. Renewed 27.7.26).—A tube passing through a crucible at a substantial distance below the axis of the periphery of the crucible, co-operates with the sides and bottom of the crucible to form a closed channel opening into the crucible on opposite sides.

J. S. G. THOMAS.

Electrolytic rectifier. C. E. OGDEN, Assr. to KODEL RADIO CORP. (U.S.P. 1,661,703, 6.3.28. Appl., 6.8.26).—An electrode containing silicon is used in conjunction with an acid electrolyte containing sulphuric acid (d 1.3—1.7).

J. S. G. THOMAS.

Electric incandescence lamp and manufacture of its illuminating body. F. SKAUPY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,659,749, 21.2.28. Appl., 2.12.21. Ger., 4.12.20).—Light radiators of the type described in B.P. 187,613 (B., 1923, 542 A) are used and heated by passage of current through a metallic conductor.

T. S. WHEELER.

Helium space-discharge tube. F. S. McCULLOUGH (U.S.P. 1,661,436, 6.3.28. Appl., 3.4.24).—A cathode, on which there is a substance from which helium is liberated when heated, and an anode are assembled in a tube and degassed by baking. After the tube is sealed a current is passed through the cathode whereby helium is evolved in the tube.

J. S. G. THOMAS.

Production of galvanic coatings of varying thicknesses. A. L. MOND. FROM BERNDORFER METALLWARENFABR. A. KRUPP A.-G. (B.P. 287,402, 14.9.27).—A jet of electrolyte from which metal is to be deposited electrolytically is directed on to the non-submerged surface of the article to be coated, and the thickness of metal deposited at any part is controlled by adjustment of the strength, speed, and size of the jet of electrolyte and the duration of its impingement upon the part.

J. S. G. THOMAS.

Brush for generators etc. A. A. SCHUPP, Assr. to FREDERICKSEN Co. (U.S.P. 1,664,708, 3.4.28. Appl., 2.4.24).—An alloy of lead and copper cast in equal proportions is claimed.

H. ROYAL-DAWSON.

Production of galvanic metal coatings. F.

KIRSCHNER (U.S.P. 1,664,750, 3.4.28. Appl., 12.10.23. Austr., 10.6.23).—See B.P. 217,876; B., 1925, 176.

Non-magnetic alloys (B.P. 287,236). **Enamelled wires** (B.P. 274,079). **High-resistance alloys** (B.P. 285,565).—See X. **Purification of water** (B.P. 287,196).—See XXIII.

XII.—FATS; OILS; WAXES.

Relation between constants of butter fat. O. LAXA (Ann. Falsif., 1928, 21, 97—98).—The Reichert-Meissl (*RM*), Wauters-Polenske (*WP*), and iodine values (*i*) of butter fat added together and divided by the refractive index (*n*) gave, for Czecho-Slovakian butter fats, a nearly constant value. Nine February samples gave an average value of 1.36 (1.29—1.43); 18 March samples, 1.4 (1.36—1.51); 19 April samples 1.45 (1.33—1.56); 15 May samples 1.4 (1.3—1.5); and 15 June samples 1.46 (1.34—1.6). The average mean for 82 samples was 1.4, and the equation $RM + WP + i/n = 1.4$ can be used for finding any one of the values.

D. G. HEWER.

Determination of the degree of rancidity in butter. G. S. INIKHOV and A. F. SHOSHIN (Trans. Vologda Dairy Inst., 1926, Bull. 64, 161—174).—The butter (1 g.) is mixed with light petroleum (10 c.c.) and Schiff's reagent (1 c.c.); after 10 min. the intensity of the colour is compared with that of standards prepared from formaldehyde.

CHEMICAL ABSTRACTS.

Butter-fat of colostrum. K. A. KUROCHKIN (Jaroslav Zootech. Exp. Stat. (1926), 1927, 142—144).—Colostrum fat obtained (by mixing 200 c.c. of top milk with 600—800 c.c. of hydrochloric acid, d 1.12, keeping at 90—95° for 1.5 hrs., cooling, and washing the fat with water not above 40°) 3.5 and 48 hrs., respectively, after delivery has m.p. 38.6, 28—29°, Reichert-Meissl value 19.65, 28.4, saponif. value 213.3, 226.5. The properties of colostrum fat approach those of lard; during the first 24 hrs. the colour is deep orange.

CHEMICAL ABSTRACTS.

Determination of the iodine value [of fats] by means of thiocyanate. H. STADLINGER (Pharm. Ztg., 1928, 73, 340—342; cf. Kaufmann, B., 1926, 165, 447).—The employment of solutions containing free thiocyanogen for the determination of unsaturation values of fats is described and discussed. The determination is carried out by addition of excess of the reagent solution to a weighed quantity of the fat, keeping it in the dark for 24 hrs., and determining the excess of reagent by addition of potassium iodide and titration of the liberated iodine. The results are expressed in terms of iodine, and are thus comparable with the ordinary iodine values. Since thiocyanogen adds directly to one double linking, does not react with a triple linking, and adds only to one double linking when two or more are present in the molecule, comparison of the ordinary iodine value with the "thiocyanate iodine value" gives a method of determining the nature and quantities of the various unsaturated compounds present. Applications of the method are discussed.

S. I. LEVY.

Fluorescence under the analytical quartz lamp of lard containing paraffin. E. FEDAR and L. RATH

(Z. Unters. Lebensm., 1927, 54, 321—330).—The behaviour of lard in the ultra-violet light of the quartz lamp is a valuable indication of its purity. Pure lard obtained by rendering shows no special fluorescence, but the commercial product exhibits white, green, or blue luminescence. This phenomenon may be produced in pure lard by treatment with steam, alkali carbonate, alkaline-earth hydroxides, animal charcoal, or fuller's earth. Some commercial lards showed a peculiar and intense blue-violet iridescence on the surface, and this was found to be associated with a relatively high unsaponifiable fraction. The active substance was isolated by heating the unsaponifiable residue with concentrated sulphuric acid at 105° for 1 hr. and extracting the undestroyed substance with light petroleum. The paraffin obtained on evaporation of the solvent showed a strong blue luminescence in ultra-violet light, and a trace added to a non-fluorescing lard imparted to it the fluorescence characteristic of the above-mentioned samples. The origin of the paraffin in the lard could not be regarded as accidental, and was undoubtedly due to adulteration with "white grease," which had been denatured with petroleum distillate. W. J. BOYD.

Utilisation of acid sludge from oil distillates treated with fuming sulphuric acid. K. LUKHUSHIN (Azerbejdjanskoye Neft. Khoz., 1927, [8—9], 84—88).—A study of the use of mixed acid sludge from the treatment of "vaseline" oil with fuming sulphuric acid for the splitting of animal and vegetable fats.

CHEMICAL ABSTRACTS.

Limited splitting of natural drying oils and the so-called non-drying linseed oil. A. EIBNER and R. HELD (Chem. Umschau, 1928, 35, 65—77).—Earlier work demonstrated that the rise of acid value of films with time varies for the different drying oils. Thus, in 11 days the acid value of a sample of poppy-seed oil rose from 7.7 to 100, linseed oil from 3.04 to over 57, whilst China-wood oil changed more slowly and from 3.7 rose to 7—13 in the same period. After 127 days the poppy-seed oil had an acid value of 221 and the wood oil 62. Determination of this value affords a method of tracing the course of the reactions in film formation. Experiments are described on the artificial partial hydrolysis of fatty drying oils by the action of water under pressure. Linseed oil (25 g.) was heated with 1 g. of water in bomb tubes at 110°, 150°, 200°, and 250° for 70, 55, 36, and 24 hrs., respectively. The resulting oils had acid values 7.07, 64.3, 72.5, and 73.5, whilst the iodine values and mol. wt. had fallen but slightly, 176.5 to 147.5 and 701 to 524 for the oils undergoing the first and fourth treatments respectively. The natural and artificial processes are compared. From the crude products were isolated, purified, and determined: α - and β -linolenic, α - and β -linoleic, oleic, and some solid acids. No selective splitting of the solid or liquid acids was apparent. Linseed oil prepared in various ways from partially fermented seeds was similarly examined, but it was found that the fission products resulting in the drying process contained, under these conditions, as much as 14% of oxy-acids. The mechanism of the drying of fatty-oil films, and their uses in technology, are discussed on the basis of these results.

E. HOLMES.

Cottonseed. M. T. HARRINGTON (Texas Agric. Exp. Stat. Bull., 1928, [374], 19 pp.).—A comparative study of 73 varieties. The oil content is not related to the weight or size of the seed. The characteristics of the oil do not vary. The protein content of the oil- and water-free meats is highest in the seed having the highest oil content.

CHEMICAL ABSTRACTS.

Extractor for the preparation of oat and other cereal oils. L. A. MUNRO and D. S. BINNINGTON (Ind. Eng. Chem., 1928, 20, 425—427).—The extractor described is useful for extraction of large quantities of material of low oil content, and oat oil prepared in it was found to be entirely unoxidised, although of high acid value, probably owing to hydrolysis which could be eliminated by immediate extraction after cracking the oats. The apparatus consists of a 20-gal. drum, containing solvent and extracted oil, provided with a gauge and heated by 2-in. steam-pipes; an extraction chamber 2 × 3 × 3 ft. with false bottom, with a false top which has $\frac{1}{2}$ -in. perforations, flanged upwards, to ensure even distribution of the solvent; and a condenser fitted with $\frac{3}{8}$ -in. copper radiator pipes. The three chambers are placed one above the other, and by suitable manipulation of valves the solvent may be first prepared, the extraction made until a concentration of about 60% of oil is obtained, and, after clarification, the oil concentrated by vacuum distillation.

D. G. HEWER.

Fat emulsions for leather. SCHINDLER.—See XV.
Detection of cacao butter. COLOMBIER and CHAIZE.—See XIX.

PATENTS.

Production of commercial stearic acid. J. STARRELS (U.S.P. 1,659,790, 21.2.28. Appl., 15.10.23).—Crude fats containing free fatty acids are heated with ethyl alcohol and an esterification catalyst, and the ethyl esters obtained are washed with water, distilled *in vacuo*, hydrogenated, and hydrolysed.

T. S. WHEELER.

Manufacture of soaps. P. FRIESENAHN (B.P. 285,174, 19.11.26).—Ordinary soap is compounded with a mixture of higher alcohols, *e.g.*, cyclohexanol, and aromatic or hydroaromatic sulphonic acids, *e.g.*, naphthalenesulphonic acid or tetrahydronaphthalenesulphonic acid, or their sodium salts.

F. R. ENNOS.

Treatment of solid material for extraction of water, oil, and fatty matter therefrom. J. R. STERLING (B.P. 286,752, 8.11.26).—Animal or vegetable material to be extracted is subjected to lixiviation with a suitable solvent on the contra-flow principle in described apparatus.

S. S. WOOLF.

Apparatus for separating and recovering oil from an emulsion of oil and water. G. B. ELLIS. From AMER. SHEET & TIN PLATE Co. (B.P. 287,438, 13.9.26).—Alkaline emulsions of palm oil and water obtained from the tin-plating process are separated, and the oil is recovered by treatment in a specially-designed apparatus with a small quantity of sulphuric acid. The mixture is agitated and heated by means of jets of live steam, and, after separation, the oil is finally dehydrated by means of a centrifuge.

C. O. HARVEY.

Reaction products of cashew nut-shell oil. M. T. HARVEY (B.P. 259,959, 11.10.26. U.S., 16.10.25).—The iodine value of cashew nut-shell oil is reduced by acid oxidation or other method and the resulting gum, dissolved in suitable solvents, *e.g.*, amyl acetate, light petroleum, etc., is used in paints, varnishes, moulded compositions, waterproofing and insulating materials, etc. S. S. WOOLF.

Alkylated fats and fatty acids (B.P. 286,796).—See III.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Influence of adsorption on the oil absorption of pigments. H. GROHN (Farben-Ztg., 1928, 33, 1660—1664).—The work of Gardner, Wagner, Klumpp, and others on oil absorption of pigments is summarised. A series of experiments on the absorption of oil, water, petroleum spirit with and without the addition of linseed oil fatty acids, stored and freshly-distilled turpentine, and 10% aqueous saponin solution by a range of pigments is described, and the results are considered in the light of Gurwitsch's views on adsorption. It is shown that the "vehicle-absorption" of a pigment depends on the nature of the vehicle, the nature and particle size of the pigment, and on their mutual adsorption capacities. S. S. WOOLF.

Testing of lithopone. ANON. (Farben-Ztg., 1928, 33, 1657).—Standard methods for the examination of lithopone are detailed under the headings:—moisture, total zinc, zinc oxide, sieving, oil absorption, fastness to light, colour, opacity, hiding power, water-soluble matter, behaviour in spirit varnish, and method of extraction from oil pastes. S. S. WOOLF.

Shellac. W. NAGEL and (Frl.) M. KÖRNCHEN (Wiss. Veröff. Siemens-Konz., 1927, 6, 235—275).—A monograph embodying the most recent contributions to knowledge of the composition and properties of shellac. The sources, commercial varieties, and industrial treatment of the material are dealt with in detail. S. I. LEVY.

Albertol resins. H. WOLFF [with W. TOELDT and G. ZEIDLER] (Farben-Ztg., 1928, 33, 1724—1727).—Albertols compare favourably with kauri and Congo copals, and are decidedly superior to ester gum when each is made up into a standard oil-varnish. Properties studied were elasticity, tensile strength, drying, yellowing, and water-resistance. S. S. WOOLF.

Drying and non-drying oils. EIBNER and HELD.—See XII.

PATENTS.

Manufacture of lacquers, impregnating materials, etc. W. CARPMAEL. From I. G. FARBENIND. A. G. (B.P. 286,789, 10.12.26).—Cellulose esters or ethers are dissolved in strongly aqueous cyclic acetals derived from polyhydric alcohols, with or without other solvents and the usual additions. Examples are cellulose acetate (10 pts.), with water (30 pts.) and monoformaldehyde-glycerol (60 pts.), or with water (20 pts.) and formaldehyde-glycol (70 pts.). These lacquers may be applied with an unusually large proportion of added water. C. HOLLINS.

Manufacture of lacquers. K. SCHMIDINGER (U.S.P. 1,663,733, 27.3.28. Appl., 1.7.25. Ger., 13.5.25).—A benzol solution of resin is saponified with alcoholic potash, the resulting soap separated from the solution of unsaponifiable material, and the latter isolated by evaporating the solvent. F. G. CLARKE.

Nitrocellulose composition. Dibutyl mesotartrate. C. BOGIN, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,659,906—7, 21.2.28. Appl., [A] 14.8.25, [B] 26.1.27).—(A) *n*-Butyl mesotartrate is claimed as a plasticiser for nitrocellulose lacquers. (B) The compound has m.p. 49°, b.p. 205°/20 mm. T. S. WHEELER.

Production of a binding material for colours, varnishes, and cements. J. TENGLER (U.S.P. 1,660,851, 28.2.28. Appl., 14.5.27. Switz., 11.5.26).—Waste rubber is heated under pressure with tetralin to remove sulphur as hydrogen sulphide, the product is dissolved in benzene, and the solution cooled to separate naphthalene and treated with sulphur to revulcanise the rubber. T. S. WHEELER.

Thinning of varnish. A. L. BROWN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,660,914, 28.2.28. Appl., 4.10.24).—The hot varnish is treated with turpentine at 175°, and when cold, with a turpentine substitute which need not be free from sulphur. T. S. WHEELER.

Manufacture of coating compositions containing rubber and drying oils. [A] C. M. STINE, [B] C. M. A. STINE and C. COOLIDGE, [C] C. COOLIDGE and H. E. EASTLACK, Assrs. [A—C] to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,663,100—1 and 1,663,119, 20.3.28. Appl., [A] 19.12.23, [B] 5.9.24, [C] 3.7.25).—(A) A mixture of a solution of rubber (10 pts.) in a rubber solvent, with a drying oil (4—31 pts.), a drier (0.4—5% of the oil), sulphur (not exceeding 0.2 pt.), and an accelerator of vulcanisation is used. Thin films are heated at between 70° and 125° to evaporate the solvent, oxidise the oil, and vulcanise the rubber. (B) The solution of rubber (10 pts.) mixed with a drying oil (30—80 pts.) is thinned with a volatile solvent to a suitable consistency, and a cobalt drier added equivalent to 0.001—0.01% Co on the oil used; the viscosity of the composition on exposure to air remains satisfactory for more than 2 days. (C) The viscosity of a coating composition containing a rubber solution and a drying oil is regulated by the addition of 0.05—10% of oleic acid. D. F. TWISS.

Manufacture of condensation products of urea and formaldehyde. I. G. FARBENIND A. G. (B.P. 258,289, 13.9.26. Ger., 11.9.25).—Carbamide and formaldehyde are condensed in an aqueous solution the pH value of which is maintained at 4—7 by the use of suitable buffer compounds, *e.g.*, potassium oxalate, mono- or di-sodium phosphate, etc. Water is subsequently removed from the condensation mass by evaporation below 50°, preferably *in vacuo*, and the product is hardened at 50—100°. S. S. WOOLF.

Continuous preparation of phenolic resins. V. H. TURKINGTON, Assr. to BAKELITE CORP. (U.S.P. 1,660,403, 28.2.28. Appl., 20.2.24).—A mixture of phenol and aqueous formaldehyde solution is passed under pressure through a coil immersed in an oil-bath maintained by

heating or cooling at a suitable elevated temperature, and the product is discharged into a vacuum chamber in which the resin is dried. T. S. WHEELER.

Grinding or mixing of paints, inks, etc. J. BAXENDALE, and HUGHES & TRELEAVEN, LTD. (B.P. 287,658, 11.1.27).

Cashew nut-shell oil (B.P. 259,959).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Vulcanised "racking" of masticated rubber. Anisotropic vulcanisation. H. FEUCHTER (Kautschuk, 1928, 4, 48—51; cf. Wiegand and Braendle, B., 1923, 412 A).—Vulcanised rubber is easily distinguished from unvulcanised sheet by the "racking" phenomena possible with the latter. A sheet of rubber showing "calender grain," however, still exhibited this feature after vulcanisation with sulphur chloride vapour and warming to 100°; swelling with benzene occurs anisotropically, but, after drying, the anisotropic character persists in the vulcanised sheet. D. F. TWISS.

Measurement of resistance of vulcanised rubber to penetration of benzene and other combustible substances. F. C. SCHMELKES (Ind. Eng. Chem., 1928, 20, 430—431).—A piece of the hose to be tested is surrounded by the liquid in question and air is passed through at a standard rate. The issuing air is subjected to continuous analysis for hydrocarbon vapours by a calorimetric device described earlier (Lamb and Larson, B., 1920, 132 A), combined with a sensitive recorder. For calibration of the instrument, carbon monoxide is advantageous. D. F. TWISS.

Diffusion researches with rubber solutions. D. KRÜGER (Gummi-Ztg., 1928, 42, 1471—1474).—The diffusion coefficient for various grades of rubber was investigated by allowing a layer of solution to diffuse upwards into a superposed column of solvent for a number of days, then dividing the whole column into four layers, and determining the rubber content of each. The value of the diffusion coefficient for any one sample is greater the higher the concentration and the shorter the period, thus indicating either that rubber is a mixture of substances of different mol. wt. or that the rubber substance is present in various degrees of aggregation. Two components indeed may be present, one easily diffusible and the other sparingly so. Crêpe, smoked sheet, and Para rubbers give substantially equal diffusion coefficients. The diffusion coefficient is much smaller in hexane and ether than in benzene; calculation of the molecular magnitude by the formulæ of Einstein and Euler gives somewhat discordant results, but indicates a much lower mol. wt. in benzene. Mastication also reduces the molecular magnitude of rubber. D. F. TWISS.

Prevention of scorching during the mixing of rubber "compounds." W. ESCH (Kautschuk, 1928, 4, 51—55).—Although generally the tendency to scorching is greater the more powerful the accelerator of vulcanisation used, mixtures containing diphenylguanidine are more prone to scorching than mixtures with the more active di-*o*-tolylguanidine. Softening agents such as stearic acid or lead oleate reduce the tendency to scorching. Lead dioxide is particularly

effective in checking scorching, the quantity necessary (ranging from 1% downwards on the rubber) varying for different accelerators. D. F. TWISS.

Direct determination of rubber in soft vulcanised rubber. A. R. KEMP, W. S. BISHOP, and T. J. LACKNER (Ind. Eng. Chem., 1928, 20, 427—429; cf. Kemp, B., 1927, 372).—After successive extraction with acetone and chloroform, a sample (0.07—0.10 g.) is dissolved by refluxing with 50 c.c. of tetrachloroethane in an oil-bath at 160—170° for 3—5 hrs., a blank experiment being made in parallel. The cooled solution is diluted with 25 c.c. of purified carbon disulphide and 25 c.c. of 0.2*N*-Wijs solution are added. After 2 hrs. in diffused light, 25 c.c. of 15% potassium iodide solution are introduced and 50 c.c. of water, the liberated iodine being then titrated with 0.1*N*-thiosulphate. Sulphur combined with rubber is determined independently by dissolving 0.5 g. of the acetone- and chloroform-extracted rubber in tetrachloroethane, diluting to 250 c.c. with carbon tetrachloride, and allowing to settle; 100 c.c. of the clear liquid are evaporated to dryness and the sulphur is determined in the residue. The rubber content is calculated on the assumption that pure caoutchouc has an iodine value 372.8, and that 1 g. of combined sulphur corresponds with 2.13 g. of caoutchouc. The influence of carbon black, glue, cellulose, common inorganic compounding materials, and, in most cases, mineral rubber is negligible; the presence of factice in quantities exceeding 1% calls for a correction. The process is more accurate and more rapid than the customary indirect method. D. F. TWISS.

PATENTS.

Vulcanisation of rubber. GOODYEAR TIRE & RUBBER Co., Assees. of L. B. SEBRELL (B.P. 265,920, 16.11.26. U.S., 12.2.6).—Substitution or additive derivatives of 1-mercaptobenzthiazoles (excluding halogen derivatives) are claimed as vulcanisation accelerators. Examples are 5-nitro-1-mercaptobenzthiazole, its zinc and lead salts, the corresponding amine, and its benzaldehyde condensation products. C. HOLLINS.

Accelerators for the vulcanisation of rubber. E. C. R. MARKS. From RUBBER SERVICE LABORATORIES Co. (B.P. 286,749 and 287,001, 6.10.26).—(A) The accelerative effect of thiol accelerators, *e.g.*, mercaptobenzthiazole, is improved if these are combined chemically with an organic base, *e.g.*, hexamethylenetetramine, phenyldiguanide, triphenyldiguanide, or guanilylurea. The two components may be heated together. (B) The reaction product of a thiol accelerator and an organic base, such as a disubstituted guanidine, is combined with an aldehyde, *e.g.*, crotonaldehyde or heptaldehyde; the resulting resinous compounds are easily dispersed in rubber. D. F. TWISS.

Rubber vulcanisation accelerator. C. O. NORTH and W. SCOTT, Assrs. to RUBBER SERVICE LABORATORIES Co. (U.S.P. 1,664,481, 3.4.28. Appl., 17.12.24).—The compound produced by the interaction of hydrogen sulphide and the ammonia addition product of a straight-chain aldehyde is claimed. F. G. CLARKE.

Manufacture of coloured vulcanised rubber. E. R. BRIDGWATER, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,663,436, 20.3.28. Appl., 29.1.26).—Rubber

or rubber-like material is mixed with a vulcanising agent and with the sodium salt of the dye obtained by coupling diazotised 2-chloro-5-toluidine-4-sulphonic acid with β -naphthol. D. F. TWISS.

Treatment of rubber emulsion to produce an adhesive. JOHNSON & JOHNSON (GT. BRITAIN), LTD. From JOHNSON & JOHNSON (B.P. 286,527, 15.6.27).—Rubber latex is diluted with a weak solution of a gum, e.g., gum tragacanth. The more concentrated upper layer which forms as a cream is further purified by shaking with an aqueous solution of an alkaline substance, e.g., sodium silicate, and of a germicide, e.g., formaldehyde, and again caused to separate by the addition of gum tragacanth. It is then mixed at 70° with one tenth of its volume of a solution containing 5% of agar and 1% of sodium silicate; the resulting emulsion, which is fluid while hot and gelatinous when cold, is squeezed in the latter state through fine gauze so as to impart a pasty consistency. The product is especially of use as an adhesive for attaching bandages and surgical dressings. D. F. TWISS.

Manufacture of articles from ebonite and like material. A. FRASER, and RISSIK, FRASER, & Co., LTD. (B.P. 286,915, 2.11.26 and 26.5.27. Cf. B.P. 273,355; B., 1927, 789).—Ebonite dust is subjected to heat and pressure inside moulds, e.g., of iron or steel, the inner surfaces of which are plated with a metal, e.g., tin or chromium, or an alloy which resists adhesion or chemical reaction with the dust. D. F. TWISS.

Manufacture of [rubber] stuffing. G. J. MILLER (U.S.P. 1,663,852, 27.3.28. Appl., 14.6.27).—Scrap rubber is softened by soaking it in kerosene until it is easily ground and, after grinding, is permanently softened to the required extent by treating it with kerosene at below 200°. The oil is separated from the mass, which is then dried. F. G. CLARKE.

Manufacture of tubes of indiarubber or similar material. DUNLOP RUBBER Co., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 287,592, 21.10.26).

Disulphide compounds (B.P. 277,338).—See III. **Binder for varnishes** (U.S.P. 1,660,851). **Coating compositions** (U.S.P. 1,663,100—1 and 1,663,119).—See XIII.

XV.—LEATHER; GLUE.

Avaram bark. I. K. S. CHOUDARY and E. YOGANANDAM (J. Soc. Leather Trades' Chem., 1928, 12, 53—58).—Avaram bark (*Cassia auriculata*), or "tarwad," is not very rich in tannin. It contains tans 17.6%, non-tans 10.8%, moisture 9.1%, and insoluble matter 62.5%. Samples from different districts vary slightly in tannin content. The tannin content increases with age from 15.58% to 19% in five years, but the ratio of tans to non-tans is fairly constant. The amount of tannin extracted at different temperatures increases with the temperature (optimum 95—100°). Of the total extractable tannin, 75% is extracted at ordinary temperatures (35—40°). The ratio of tans to non-tans does not vary much with the temperature of extraction. The colour of the leached liquors is deepest at low temperatures of extraction and becomes brighter at higher temperatures. The p_H value of both avaram and

komman (*Cassia fistula*) liquors is 4.7. Avaram bark liquors do not penetrate so well as liquors prepared from other South Indian tanning materials. Avaram liquor kept for 125 days lost but little tannin, whereas the non-tans diminished by 6.8%; thus, the ratio tans/non-tans fell from 1:1.11 to 1:0.53. Avaram liquors lose least tannin of all the South Indian materials on keeping. Avaram-tanned leather is mellow and very light coloured, but darkens quickly on exposure to light. Most of the leather exported from Southern India is lightly tanned. An unadulterated sheep skin tanned with avaram had water-soluble matter 8.0%, fat 9.85%, moisture 12.5%, and leather substance 69.65%. The loss on washing was 5.5%. Spent avaram bark from the first, second, and third liquors, respectively, contained 5.87, 7.02, and 9.35% of tan. D. WOODROFFE.

Microscopical examination of fat emulsions used in the leather trade. W. SCHINDLER (Collegium, 1928, 12—20).—Several emulsions of oils and fats at different p_H values, respectively, have been examined microscopically. Soap (10 pts.) and mineral oil (1 pt.) creamed at p_H 9—10 but remained partly emulsified at 8.6. A mixture of 20 pts. of Turkey-red oil and 1 pt. of mineral oil gave an opalescent emulsion which at p_H 7.2 showed no globules under the microscope, but at p_H 6.8 tiny globules became apparent; the emulsion was milky but stable. At p_H 6.1 there were much larger globules and the liquor creamed. A number of sulphonated fish oils have been similarly examined. A sulphonated neat's-foot oil was stable for 30 min. at p_H 7.9, but separated quickly below this value, and an addition of mineral oil did not improve the stability of its emulsions. Fresh egg-yolk emulsion was creamy and consisted of very small separate globules. At p_H 7.8 the emulsion with mineral oil was opalescent and creamed after being kept several hours; there were no groups of fat globules. The emulsion was more permanent at p_H 6.1. Soap-degrad emulsions creamed at p_H 9.5, 8.95, and 8.7. The results showed that the formation of groups of globules was indicative of emulsions which creamed. Increase of p_H value improves the colour and diminishes the number of visible globules, but does not always make the large drops smaller. These results confirm those of Stiasny (B., 1925, 965) and Riess. The emulsifying oils did not reduce the mineral oil to globules of definite size. Sulphonated oils had very little effect on the surface tension of the mineral oil to water, whilst they split up the mineral oils into very small globules and gave a fairly stable emulsion, which was less sensitive than the soap emulsions. The emulsifying power of sulphonated oils acting on mineral oil appears to depend on the ratio $(\alpha + \beta) : \gamma$. The amount of mineral oil affects the emulsifying power, and in commercial products is often considerable. Since α may be considered to consist of fatty acids and will disappear at p_H 6.1—7.0, it is evident that β is the important factor. The hydroxy-acids in β are less effective than the sulpho-fatty acids, so that the emulsifying power ultimately depends on the latter. D. WOODROFFE.

Effect of salts and temperature on the manufacture of enzyme [bating] extracts and the results of enzyme analyses. J. SCHNEIDER, JUN., and A.

Určák (Collegium, 1928, 22—26; cf. B., 1927, 662, 758).—Enzyme extracts were prepared from "Oropon-standard" and different amounts of various salts by extracting for 30 min. at 18°. The filtered extract (50 c.c.) was tested on casein solution. Increasing amounts of ammonium sulphate, ammonium chloride, sodium chloride, ammonium and sodium chlorides, and ammonium sulphate and chloride caused increased activity of the enzyme extract. Curves have been obtained for the effect of these salts on the activity of the enzyme extract. Extractions at different temperatures showed maxima at 24° in two experiments, at 32° in a third, and a diminution of activity with rising temperature in two other tests.

D. WOODROFFE.

Effect of hydrogen-ion concentration on tannage.

P. PAVLOVITSCH (Collegium, 1928, 2—12).—A 1% solution of gelatin was mixed with 2—3 pts. of a 1% solution of quebracho, and the amount of the precipitate determined for different p_H values, respectively. Maximum combination took place at p_H 2; the amount of combination is practically uniform from p_H 3 to 6; at p_H 7.0 it was less and at p_H 8 very small. Further experiments with hide powder showed a steep fall in the combination curve at p_H 7.5—8.0, but no maximum as suggested by Thomas and again by Wilson (cf. Thomas and Kelly, B., 1923, 1235 A). Sole-leather pelts are penetrated most quickly at p_H 7—8. The penetration at 35—37° is 2—3 times as great as that at 15—20° using quebracho liquors of d 1.091. Oakwood liquors (d 1.091) penetrated best at p_H 6. Valonia liquors showed a more pronounced maximum fixation of tannin from acid liquors than oakwood. Mimosa, mangrove, and chestnut liquors (d 1.091) gave best penetration at p_H 8, 7, and 3—4, respectively. Tannage must be commenced with liquors of minimum astringency, the p_H values of which are as follows: (at d 1.091) quebracho 7—8, oakwood 6—7, mimosa 8, mangrove (d 1.60) 4, valonia extract ("Valex") 4, and chestnut 3—4. Pelts can be tanned in these strong liquors at these respective p_H values. It is not necessary, as Thomas states, to commence the tannage at the isoelectric point of collagen (p_H 5.0). Alkali should be added to strong tan liquors to increase the p_H value to 6—8, the pelts should be well penetrated by this, and then the p_H value should be lowered gradually to 3—4. The leather can afterwards be retanned in strong extracts. This process will not cause "dead tannage" or drawn grain. These various phenomena can be explained not only by the degree of dispersion of the tannin particles, but also by dehydration and rehydration. By combining the rehydrating medium at the commencement of tannage, the tannins can be forced to penetrate the pelt without tanning, and then, by dehydrating, the tannins can be coagulated and fixed on the hide substance. Colloids which have been dehydrated by tannin can be rehydrated by raising the temperature, adding organic substances such as non-electrolytes, e.g., alcohol, glycerin, glycol, or electrolytes such as acetic acid of moderate concentration, or by adding alkalis (cf. B., 1923, 789 A).

D. WOODROFFE.

American procedure in the colouring and finishing of leather. M. C. LAMB (J. Soc. Leather Trades'

Chem., 1928, 12, 58—63).—Most of the dyeing is done in drums, and mechanical devices are used wherever possible. The general lay-out of the works is such that there is no retrograde step. A much larger bulk of goods is handled in the different processes. American dyes are used and basic dyes preponderate. Pigment finishes or "dopes" are applied in the finishing of the leather by means of a machine fitted with a row of rotatable brushes. Spray-dyeing has not been applied to the same extent as in Great Britain. The spraying of pigment finishes is quite common. Every leather works in U.S.A. is equipped with drying machines. The skins are tautly strained by means of special clips on wire netting or perforated metal frames, which are placed in position in a drying chamber resembling a filing cabinet. Two men with a dozen sections of such a machine, capable of drying 2 doz. skins at a time, can be continuously occupied throughout the day. The actual drying requires a comparatively few minutes. The more rapid drying ensures greater fullness of shade than slow drying.

D. WOODROFFE.

Causes of drawn grain on chrome upper leathers.

J. S. MUDD (J. Soc. Leather Trades' Chem., 1928, 12, 49—52).—The waste liquors from chrome tannages which had caused drawn grain showed p_H values less than 3.7. Liquors buffered with sodium acetate to give p_H 4.3—4.9 gave a quicker tannage free from drawn grain. Other tests in which the p_H value decreased during the tannage produced drawn grain. It is suggested that acid swelling at p_H values 4.0 or above 5.0 will cause "pebbling" or drawn grain.

D. WOODROFFE.

Centrifuging as an aid to tannin analysis by the shake method.

V. KUBELKA (Collegium, 1928, 21—22).—To avoid the use of linen cloths, the operations in tannin analysis are performed in a centrifuge tube (200 c.c.) fitted with a rubber stopper. The hide powder can be shaken in the tube with water, chroming solution, wash water, or tannin infusion. Centrifuging at 4000 r.p.m. for 5 min. effectively separates the clear liquor.

D. WOODROFFE.

Cleaning leather. GOLDMAN and HUBBARD.—See V.

PATENTS.

Treatment of the skins of sharks and other fish of the same kind. A. EHRENREICH (B.P. 284,197, 24.3.27. Belg., 24.1.27).—The dermal armour is removed by treating the skins before or after tanning with a solution of sodium chloride and phosphoric or hydrofluoric acid, with or without an alkali salt of the acid, or aluminium salts, and/or small quantities of metallic salts which act as catalysts. An electric current may be passed through the solution, which is circulated by jets of compressed air.

D. WOODROFFE.

Derivatives from ligninsulphonic acid (B.P. 286,808).—See III. **Dyeing of fur** (U.S.P. 1,660,826).—See VI.

XVI.—AGRICULTURE.

Availability of potassium to plants as affected by barnyard manure. R. P. BARTHOLOMEW (J. Amer. Soc. Agron., 1928, 20, 55—81).—Barnyard manure may contain 3—20 (from the same herd 5—12) lb. of potassium per ton; all of it is available for plant growth

before much of the organic matter is decomposed. Since crops remove more potassium than is added as manure, the decomposition of the organic matter of the manure may convert some of the insoluble soil potassium into an available form.

CHEMICAL ABSTRACTS.

Ammoniacal nitrogen of peats and humus soils.

II. J. C. B. ELLIS and C. G. T. MORRISON (*J. Agric. Sci.*, 1928, **18**, 346—349).—The amount of ammoniacal nitrogen obtainable from bog peat by distillation under reduced pressure with magnesia increases with the extent of the previous drying of the sample. This is attributed to a reduction of the colloidal adsorptive power of peat during drying. Subsequent moistening of dried peat allows of a partial recovery of its adsorptive properties. (Cf. B., 1916, 1167.)

A. G. POLLARD.

Time of using nitrogenous fertilisers on winter crops. E. MÖLLER-ARNOLD and E. FEICHTINGER (*Z. Pflanz. Düng.*, 1928, **7B**, 119—127).—On light and medium soils better results were obtained with winter wheat and rye by adding the whole amount of the nitrogen fertiliser in spring than when used part in autumn and part in spring. On stiff clays it was preferable to apply the whole of the nitrogen fertiliser in autumn. With rye on sandy soils, the increased yield obtained by a complete nitrogenous dressing in spring as compared with a divided dressing in spring and autumn, was mainly in the straw. In this respect the efficiency of a unit of nitrogen added in spring was five times that of a unit added in autumn when calculated from a Mitscherlich growth-curve.

A. G. POLLARD.

Influence of calcium cyanamide on the bacterial life of soils. J. KUHN and O. DRECHSEL (*Z. Pflanz. Düng.*, 1928, **7B**, 105—118).—Calcium cyanamide causes considerable increases in the bacterial numbers of soils for a period of 2—3 months from its use. This effect is specific to "nitrolim," and is not brought about by free cyanamide either alone or mixed with lime. It is not the result merely of treatment of soil with lime and nitrogen fertiliser. Sodium cyanamide produces relatively small increases in bacterial numbers. The stimulation of bacterial growth following the use of "nitrolim" is greater in neutral and alkaline soils than in acid ones.

A. G. POLLARD.

Sulphur in relation to the soil solution. W. L. POWERS (*Univ. Calif. Pub. Agric. Sci.*, 1927, **5**, 119—170).—The application of sulphur and sulphates to Madera sand soil increased the bases, especially calcium, in the soil solution. Heavy applications of sulphur increased the acidity, at first increasing the iron and phosphate content of the solution, then decreasing it, and inhibiting nitrification. Normal application (100 lb. per acre) increased growth and the nitrogen content of arid soil. Application of sulphur may produce a more concentrated soil solution and thus decrease transpiration.

CHEMICAL ABSTRACTS.

Chemical changes in dusting mixtures of sulphur, lead arsenate, and lime during storage. H. S. SWINGLE (*J. Agric. Res.*, 1928, **36**, 183—192).—A combined insecticide and fungicide dust (sulphur 80%, lead hydrogen arsenate 5%, calcium hydroxide 15%), which is commonly used in the United States, usually becomes dark grey, and eventually almost black, on storage.

This darkening is shown to be due, firstly, to a reaction between the calcium hydroxide and sulphur to form calcium sulphide and sulphite, and secondly, to the reaction of the calcium sulphide with lead arsenate, giving calcium arsenate and lead sulphide. Hot dry conditions of storage favour this change, which, however, has little or no effect on the efficiency of the mixture, the greatest amount of sulphur involved in the reactions in dusts stored for a year being less than 0.5% of the total amount present. Field experiments indicated that there was some increase in the risk of damage to foliage by the use of old samples.

C. T. GIMMINGHAM.

Root-soluble nutrients in soils and subsoils.

M. KLING and O. ENGELS (*Z. Pflanz. Düng.*, 1928, **7B**, 127—139).—Examination is recorded of a large number of surface soils and subsoils. In general, the subsoils are the poorer in root-soluble potash and phosphate, but the relative proportions in soil and subsoil vary enormously. Decrease of nutrients with depth of soil was more marked with phosphate than with potash. Extraction of soils with 10% hydrochloric acid gives no information as to the amount of root-soluble nutrients present. By the Neubauer method one third of the soils examined was deficient in potash and two thirds were deficient in phosphate. The importance of examining subsoils for nutrients, to depths varying with the nature of the root system of the growing crop, is emphasised.

A. G. POLLARD.

Zeolite formation and base-exchange reactions in soils.

P. S. BURGESS and W. T. MCGEORGE (*Arizona Agric. Exp. Stat. Tech. Bull.*, 1927, [15], 359—398).—Soil permeability is little hindered by simple silicates, but always completely retarded by aluminium hydroxide or hydrated aluminium silicate gels (zeolites) when precipitated within the soils. Sodium silicate and aluminate are usually present in the soil solution of strongly alkaline soils. When solutions of these substances are added to soils, and the latter air-dried, their base-exchange capacities are greatly augmented; addition of sodium hydroxide to certain soils has a similar effect. Synthesis of artificial zeolites from different proportions of the constituents affords the same zeolite, apparently scolecite. Zeolites are considered to be chemical compounds capable of ionising and entering into true chemical reactions. Soil organic matter appears not to possess a true base-replacement capacity, but rather the ability merely to adsorb solutes.

CHEMICAL ABSTRACTS.

Dye adsorption by hydrous alumina in soils.

II. H. CROUCHER (*J. Agric. Sci.*, 1928, **18**, 350—362).—Examination of the adsorption of a large number of dyes by hydrous alumina led to the selection of Biebrich-scarlet (for acid adsorption) and iodine-green (for basic adsorption) as suitable for adsorptive measurements of soil colloids. The presence of hydrous alumina in soils was demonstrated, and results of the examination of soils by the adsorptive process are recorded.

A. G. POLLARD.

Influence of fertilisers on the vitamin-B content of wheat. C. H. HUNT (*Ohio Agric. Exp. Stat. Bull.*, 1927, [415], 1—41).—The results suggest that acid phosphate alone or in a complete fertiliser with potassium chloride and sodium nitrate produces wheat with the

highest vitamin-*B* content. Potassium chloride and sodium nitrate together, in two years out of three, produced wheat of the lowest vitamin content. Climate causes significant variations. There was no evidence that spring wheat contains more vitamin-*B* than winter wheat, nor was any parallelism observed between the vitamin-*B* content and that of calcium and phosphorus. The reproductive history of rats is a more sensitive criterion of the vitamin-*B* content of wheat than are growth curves.

CHEMICAL ABSTRACTS.

Nutritive value of pasture. III. Influence of intensity of grazing on the composition and nutritive value of pasture herbage. I. H. E. WOODMAN, D. B. NORMAN, and J. W. BEE (*J. Agric. Sci.*, 1928, 18, 266—296).—Comparison is made of the feeding value of pasture herbage cut weekly and fortnightly. No important difference in chemical composition or digestibility was observed. Although there was a slight widening in the nutritive ratio in the fortnightly- as against weekly-cut herbage, the value was still definitely narrower than that for milk. The total production of herbage during a season somewhat favoured fortnightly cutting. This difference is to be expected to increase during unfavourable growing weather. (Cf. B., 1926, 506; 1927, 588.)

A. G. POLLARD.

Influence of soil heterogeneity on growth and yield of successive crops. T. EDEN and E. J. MASKELL (*J. Agric. Sci.*, 1928, 18, 163—185).—Results of field trials were examined mathematically with a view of correlating physical conditions of the soil and plant growth. Ploughing draught, as a measure of physical condition, showed a fairly close correlation with the germination of wheat, but not with its later development. With swedes no definite relationships were apparent. In both cases the effect of the spacing of the plants on their growth and development was of primary importance.

A. G. POLLARD.

Maize silage. II. H. E. WOODMAN and A. AMOS (*J. Agric. Sci.*, 1928, 18, 194—199; cf. B., 1924, 885).—The preparation and feeding trials of maize silage are described. Silage made in towers from the French variety of maize, *Jaune Gros du Domaine*, showed a higher digestibility and nutritive value than that from American maize or the "green fruity" oat and tare silage made in this country.

A. G. POLLARD.

Micro-Kjeldahl method for determination of total nitrogen in soils. I. POLLAK and R. DIETZ (*Oesterr. Chem.-Ztg.*, 1928, 31, 57—58).—A modified method is described which gives results in close agreement with the macro-method and for which greater rapidity is claimed. 1 g. of soil passing a 1 mm. sieve is digested with sulphuric acid in a small flask together with a drop of mercury and a mixture of copper and potassium sulphates. After digestion ($\frac{1}{2}$ —1 hr.), the contents of the flask are made up to 200 c.c. and 10 or 20 c.c. are withdrawn for distillation. After the addition of sodium hydroxide and sodium thiosulphate the ammonia is distilled in steam into *N*/70-hydrochloric acid and is titrated in the usual way.

H. J. G. HINES.

Use of the Lundegårdh apparatus for the determination of the carbon dioxide production of forest soil. D. FÉRÉZ (*Biochem. Z.*, 1928, 193, 350—

355).—The apparatus (B., 1927, 662) is simplified and cheapened without losing accuracy.

P. W. CLUTTERBUCK.

Page and Williams' method for the determination of the saturation capacity of soils. P. E. TURNER (*J. Agric. Sci.*, 1928, 18, 257—265).—In this method (B., 1925, 108) the leaching of the soil treated with calcium carbonate to a total volume of 2 litres is insufficient to remove all exchangeable bases from the soil, and should be continued to 3 litres or more. Alternatively, the size of the sample of soil should be reduced. The difference in the amount of calcium appearing in the first and second litres of extract as a result of dissolution of the calcium carbonate is negligible. The displacing reaction proceeds very slowly when nearing completion, and for this reason Hissink's method for determining saturation capacity is inaccurate. The ratio of displaced calcium appearing in the second litre of extract to the total amount is influenced to some extent by the nature of the colloidal matter present.

A. G. POLLARD.

Specific conductivities of soil extracts. C. H. WRIGHT (*J. Agric. Sci.*, 1928, 18, 186—193).—The specific conductivity of Nigerian cotton soil extracts varied considerably with the season, and tended generally to reach a maximum in March and a minimum in June-July. Comparison of the specific conductivities of different soils can only be made when samples are drawn at the same period. There was a relationship between the conductivity of 1:5 soil: water extracts and the yield of cotton lint, and the former may be taken as a measure of soil fertility. Decreased specific conductivity of soils under continuous cropping is paralleled by the decreased solubility of the soils.

A. G. POLLARD.

Apparatus for measuring the hydrogen-ion concentration of the soil. R. H. BRAY (*Ind. Eng. Chem.*, 1928, 20, 421—423).—The electrode vessel is a Gooch funnel with the stem bent up, and a perforated porcelain plate sealed into the bottom. The entering hydrogen bubbles up through the plate at a sufficient rate to keep the soil particles (10 g. of soil in 25 c.c. of distilled water) in suspension, and escapes from a hole in the stopper which also serves for the entrance of the agar-potassium chloride tube. The electrode is a piece of approximately 20-gauge platinum wire 10 cm. long, sealed into a glass tube, bent to a spiral cone, and plated with palladium-black. The agar-potassium chloride tube is connected with saturated potassium chloride solution, connected again with a saturated calomel half-cell. The electrodes must have reached equilibrium before readings are taken, and should be checked when newly plated against a standard phthalate buffer solution.

D. G. HEWER.

Determination of the p_H values of turbid soil and other solutions. C. H. GADD (*J. Agric. Sci.*, 1928, 18, 206—208).—To each of two similar portions of a 1:2 soil: water suspension decanted after being kept for 24 hrs. is added the requisite amount of indicator solution prescribed by Clark's method. To one of the samples a buffer solution of known p_H value is added, and the colour of the mixture compared with the second or control sample. The process is repeated with other

buffer solutions until no colour change (or a minimum) results.

A. G. POLLARD.

An index of soil texture. F. HARDY (J. Agric. Sci., 1928, 18, 252—256).—For soil survey purposes an "index of texture" (*I.T.*) is developed, and is evaluated thus: $I.T. = P - S/5$, where *P* is the moisture content of the soil at its point of stickiness and *S* is the sand content. Although distinguishing only main soil types, and giving no indication of organic content, the index of texture offers a single-valued and rapidly-determined figure for soil characterisation. Values obtained are closely paralleled by those of hygroscopic coefficients, the mean ratio to which for a number of soils is 4—6.

A. G. POLLARD.

Mechanical analysis of heavy ferruginous soils. R. C. GROVES (J. Agric. Sci., 1928, 18, 200—205).—The difficulty of dispersing ferruginous clays prior to mechanical analysis is overcome by a modification of the Robinson method, whereby the soil organic matter is broken up by heating with successive small quantities of ammoniacal hydrogen peroxide. The residual soil may be worked up into a suitable suspension merely by gentle rubbing with a rubber pestle and decanting in portions. Mechanical shaking is unnecessary.

A. G. POLLARD.

Soil stratometer. Examination of deep-lying soil. M. ZAGHLOUL (Nature, 1928, 121, 537).—A simple instrument for measuring variations of the mechanical resistance of soil in a vertical direction is described.

A. A. ELDRIDGE.

Analysis of insecticides. Dry products: naphthalene; naphthalene and camphor; blue powders containing strychnine; red corn. M. FRANÇOIS and (Mile.) L. SÉGUIN (J. Pharm. Chim., 1928, [viii], 7, 331—340).—Methods are given for the identification and determination of naphthalene and camphor in admixture, strychnine, brucine, and other alkaloids in presence of methylene-blue, and arsenic in blue powders, and of strychnine sulphate and colouring matters in impregnated corn.

E. H. SHARPLES.

Analysis of sugar beets. SHERWOOD.—See XVII.

PATENTS.

Production of insecticides. E. C. R. MARKS. From ASSOCIATED OIL CO. (B.P. 287,371, 4.7.27).—The products contain basic compounds extracted with sulphur dioxide from crude shale oil or other hydrocarbon oils and subsequently purified by extraction with sulphuric acid and steam distillation of the extract after neutralisation with sodium hydroxide, a refined oil containing, preferably, at least 90% of unsulphonatable residue, and an ammonium salt of a fatty acid. Inert material, e.g., kieselguhr, and a red dye may also be added, the mixture being worked up to a stable emulsion with water.

L. A. COLES.

Fungicides for seed grain and the like. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 287,246, 20.12.26).—The fungicides contain arsenious or arsenic acid or their salts and mercury compounds of phenol or its homologues, e.g., mercurinitrophenol, with or without

the addition of copper compounds or other fungicides and talc, gypsum, kieselguhr, or other inert material.

L. A. COLES.

Fungicide. H. L. KLEINHANS, Assr. to C. COOPER & Co., Inc. (U.S.P. 1,662,462, 13.3.28. Appl., 1.11.24).—A mixture of sulphur and copper carbonate so proportioned as to be miscible with water is used. The copper carbonate neutralises the acids generated by the sulphur in the soil.

F. G. CLARKE.

Insecticide (U.S.P. 1,663,883).—See XXIII.

XVII.—SUGARS; STARCHES; GUMS.

Use of the refractometer in the analysis of individual sugar beets. S. F. SHERWOOD (J. Agric. Res., 1928, 36, 41—51).—Total soluble solids in the juice from very small samples of sugar-beet pulp may be determined satisfactorily and rapidly by measuring the refractive index of a few drops of the juice squeezed from the pulp with the fingers and allowed to fall directly upon the prism of the refractometer. The determination may be delayed with safety up to 24 hrs. if the pulp is stored in air-tight containers in a refrigerator at about 12°. The sucrose present is not, however, proportional to the total solids, and it should be determined by means of the polariscope. The ratio of % sucrose in juice: % solids by refractometer is called "the apparent purity by refractometer," and is regarded as a valuable criterion for judging the comparative quality of individual beets.

J. R. NICHOLLS.

Extracts from a [sugar] laboratory notebook. J. P. OGILVIE (Internat. Sugar J., 1928, 30, 120—126).—Using Sperry's "equal-effect" method (B., 1923, 369 A) "Carboraffin," "Anticromos," "Supra Norit 5 X," "Supra Norit 3 X," "Suchar," and "Radit" were amongst the carbons found to give the best results in the decolorisation of molasses solution (*d* 1.03). Examined by the same process, the amount of decolorising earth (as used in the oil industry) necessary to produce the same effect as 1 pt. of "Superior Norit" was not found to be less than 8—9 pts. by wt.; although of low decolorising power, however, such earths may considerably assist in the production of clear and light-coloured juices or syrups. Sodium hyposulphite was shown to decompose at a rapid rate on exposure to air, and packing in small hermetically-sealed containers is advised. Lime in the form of hydrate is advocated for use in tempering cane juices, on account of its high purity and convenience of packing for shipment in paper-lined bags. A good filtration kieselguhr should have an apparent density of from 12 to 17 lb./cub. ft., should consist mainly of relatively large diatoms (preferably acicular in form), and should be free, or almost so, from amorphous particles of clay or chalk. Arsenic determinations in products used in cane and beet sugar manufacture gave the following results for As₂O₃ in grains/lb.:—limestone, 1/70—1/240; coke, 1/10; unrefined sulphur rolls, 2.0; refined ditto, 1/140; concentrated superphosphate, 4½; "Sumaphos," 1/117; ultramarine-blue, 1/5.

J. P. OGILVIE.

Calculation of purity factors for dry lead defecation at 20°. E. W. RICE (Sugar, 1927, 29, 522).—

Errors amounting to 28% are liable to be made if purity values are calculated from tables meant to be used with Mohr c.c. whilst actually the polarimetric readings are taken at 20°. A table of factors calculated for 20° to be used with the dry lead method is presented.

J. P. OGILVIE.

Control of p_{H} value in the sugar industry. BARBAUDY (Chem. et Ind., 1927, 18, 984—992).—A review.

J. P. OGILVIE.

Starch in gluten bread. FLEURY and BOYELDIEU.
Honey. FIEHE and KORDATZKI, also FIEHE.—See XIX.

PATENTS.

Batteries for continuous diffusion of sugar and the like. A. RAK (B.P. 286,125, 7.5.27).—The slices of beet move upwards through a succession of towers, and the diffusion liquid flows downwards and counter-current to the slices.

B. M. VENABLES.

Cleaning of chicle and similar gums. D. W. HOWE, E. R. PICKETT, and D. M. McBEAN, Assrs. to BEECH-NUT PACKING Co. (U.S.P. 1,664,981, 3.4.28. Appl., 13.7.25. Renewed 2.9.27).—The gum is emulsified in hot water, the emulsion is filtered, and the gum and excess water are separated.

H. ROYAL-DAWSON.

XVIII.—FERMENTATION INDUSTRIES.

Carbonyl value of wine. H. STRACHE and A. BRANDL (Z. Unters. Lebensm., 1928, 55, 50—53).—Aldehydes and ketones play a considerable part in the bouquet of wine, and the kind is more important than the quantity; a determination of the carbonyl group is of value in characterising a special kind of wine or a particular vintage. For this purpose a definite quantity of phenylhydrazine is added to the distillate from 100 c.c. of wine, and the nitrogen evolved from the uncombined phenylhydrazine after boiling with Fehling's solution is measured. The carbonyl oxygen from the wines examined varied from 0.0078 to 0.0239 g./litre, being higher from the wines with stronger aroma. The major part of the carbonyl compounds was found to be readily volatile, and after two thirds of the wine had been distilled no more came over. In the case of Muscatel and Chablis, the residue after distillation on being steam-distilled gave an additional quantity.

J. R. NICHOLLS.

Cœnological indices and reversion of malic acid in wines. L. FERRÉ (Ann. Falsif., 1928, 21, 75—84).—Malic acid is an important factor in the relation of alcohol content and acidity in wines, and frequently diminishes in wines that have been kept, whilst the lactic acid increases. If the acidity of a wine and its content of lactic acid are known, the initial acidity may be calculated, and it is considered logical to calculate all the cœnological indices to the original acidity. The proportion of tartaric, malic, and lactic acids in a wine forms a useful foundation in giving an opinion on the wine, lactic acid chiefly indicating the presence of malic acid reversion.

D. G. HEWER.

Adulteration of brandy and its detection. (A) H. ZELLNER (Z. Unters. Lebensm., 1927, 54, 389—390). (B) G. GRAFF (*ibid.*, 391—393).—(A) Statements made in

a lecture on the above subject by Graff (B., 1927, 953) are criticised. The fusel oil and ester number recommended by Graff are deprecated on the ground that the content of higher alcohols in wines and therefore in wine products varies too widely. Whilst analytical numbers are not to be despised, taste and odour are the most reliable indications that wine products so-called are genuine.

(B) A reply. It is claimed that tasting must occupy a secondary position seeing that it is subjective. Further, the addition of sugar to wine before fermentation, or of rectified alcohol before distillation, cannot be detected by taste, but can be detected by determining the amount of higher alcohols present, these being in lower proportion in wine products so adulterated.

W. J. BOYD.

Detection of cider in wine by microscopical examination of the sediment. L. MINDER (Z. Unters. Lebensm., 1927, 54, 387—388). A. WIDMER and O. E. KALBERER (*ibid.*, 388; cf. B., 1927, 568).—A question of priority.

W. J. BOYD.

Protomalt. NEUMANN.—See XIX.

XIX.—FOODS.

Glutelins of the cereal grains. R. K. LARMOUR (J. Agric. Res., 1927, 35, 1091—1120).—Alkali-soluble proteins were prepared from wheat, spelt, rice, maize, oats, einkorn, emmer, durum wheat, teosinte, rye, and barley. The general method of preparation was to treat the alcohol-insoluble grain with 0.2% alkali, to super-centrifuge, and to precipitate the soluble proteins with hydrochloric acid. The precipitate was washed with alcohol and ether, dried, and ground. The nitrogen distribution was determined by the Van Slyke method (B., 1911, 1135) whereby values were obtained for ammonia-nitrogen, total humin-nitrogen (including acid-soluble, acid-insoluble, and phosphotungstic acid-insoluble), total basic nitrogen (including arginine, cystine, lysine, and histidine), and total filtrate nitrogen (including amino- and non-amino-). The results showed well-marked relationship between the various preparations, especially in respect of the basic nitrogen fraction. Glutenin of wheat (*Triticum vulgare*) and oryzanin of rice (*Oryza sativa*) occupy positions at the extreme limits of the class in respect to ammonia and total basic nitrogen, and the corresponding values for the other proteins described fall within these limits. This is submitted as evidence that the preparations obtained belong to a definite class of proteins, the glutelins, which is represented in all the cereal grains thus far studied. A nomenclature for these substances is suggested.

J. R. NICHOLLS.

Determination of dextrose in presence of proteins; with particular reference to starch in "gluten" bread. P. FLEURY and G. BOYELDIEU (J. Pharm. Chim., 1928, [viii], 7, 207—214, 249—255).—The dextrose in a standard solution was determined by the method of Fleury and Boutot (A., 1922, ii, 879) in the presence of certain proteins and their degradation products. Albumins and amino-acids had no appreciable effect on the result, but albumoses and peptones caused a diminution in the amount of reduced copper (cf. Fringsheim and Winter, A., 1926, 1274; Neuberg and Simon, A., 1927,

450). Peptones etc. were removed therefore by treating the solution with neutral lead acetate or preferably acid mercuric sulphate solutions. The method of analysis recommended is as follows:—The dried sample of bread is hydrolysed with dilute sulphuric acid at 120° for 20–25 min. An aliquot proportion of the cooled solution is then treated with mercuric sulphate solution (200 g. of yellow mercuric oxide and 175 c.c. of sulphuric acid, d 1.83, per litre), neutralised until just acid to litmus, filtered, and the filtrate treated with zinc dust. The dextrose is finally determined polarimetrically or preferably by reduction in the new filtrate. S. COFFEY.

Addition of protomalt to rye bread. M. P. NEUMANN (Z. Unters. Lebensm., 1927, 54, 341–347).—A new bread improver for accelerating the fermentation process in rye dough has been examined. The extract known as protomalt on analysis was found to contain: water, 29.3%; protein, 6.15%; amino-nitrogen, 0.08%; nitrogen-free extract, 63.35%; reducing sugar, 43.60%; sugar reducing after inversion, 51.60%; starch, nil; ash, 1.20%; it had pH 4.9, acidity 16.2, diastatic value 17, viscosity (100 c.c. of 1:1 solution) 80 sec. (water 34 sec.). Fermentation trials showed that protomalt added to rye flour greatly promoted the development of acidity. Baking tests were carried out with leaven and with yeast, with and without protomalt. The bulkiness of the bread made with leaven was greater than that of the yeast bread, the pores were more uniform, and the results more certain. Protomalt added to the "sponge" in the yeast process gave a better bread than that obtained without it, but its addition to the dough had less satisfactory results. The addition of protomalt to the complete leaven gave a very good bread of greater bulk without the excessive browning of the crust usually found when malt preparations are used. Added to the dough it had little effect. W. J. BOYD.

Properties and uses of an edible rice cellulose. E. R. HARDING (Ind. Eng. Chem., 1928, 20, 310–311).—A pure edible cellulose may be prepared from the hulls of rice and other cereals by a strong soda boil followed by a hydration treatment, the object of which is to reduce the material to a suitable physical form. The cellulose so obtained (α -cellulose 73%, hydrated cellulose 24%, ash 0.7%) is used for raising the roughage content of many types of food. W. J. POWELL.

Detection and determination of rice flour in other flours and in spices. M. WAGENAAR (Z. Unters. Lebensm., 1927, 54, 357–369).—Microscopical and chemical methods for the detection of rice flour in buckwheat, oat, wheat, rye, and barley flours, and in pepper, mustard, mace, nutmeg, allspice, cloves, cinnamon, and cocoa are described. The microscopical appearance of the starch grains, aleurone layer, germ tissue, and the effect of various reagents on these, provide means of distinguishing particles of rice from those of other products. A quantitative microscopical count method has been devised by the author depending on the adsorption of an acid dye, *e.g.*, fuchsin-S, by the protein granules. The peculiar distribution of these in the rice grain gives a characteristic appearance to the particles of rice flour. The actual proportion of

rice present is calculated from the number and size of the rice particles in a known quantity of flour.

W. J. BOYD.

Correlation of kernel texture, test weight per bushel, and protein content of hard red spring wheat. J. H. SHOLLENBERGER and C. F. KYLE (J. Agric. Res., 1927, 35, 1137–1151).—Data from 1290 representative samples of hard red spring wheat were studied. A fairly strong tendency was noted for protein content to increase with the proportion of dark, hard, and vitreous kernels, and there was a significant correlation between test weight and protein content, the latter increasing with the former up to 54 lb. per bushel and then decreasing. The variation in protein was found to be more influenced by kernel texture than by test weight, and no correlation was observed between the latter. A method is presented for estimating the protein content when the kernel texture and test weight are known. For this purpose a table is given relating the percentage of dark, hard, and vitreous kernels with that of proteins; a second table gives the correction to be applied to the protein for the test weight.

J. R. NICHOLLS.

Solids-not-fat as criterion for watered milk. C. E. KLAMER (Z. Unters. Lebensm., 1928, 55, 45–47).—The limit of 8% for solids-not-fat in milks is stated to have no essential significance with regard to watering. The determination of the f.p. is considered to be much more reliable. For genuine milks the f.p. generally lie between -0.53° and -0.55° , although some milks give values between -0.52° and -0.53° or above -0.55° . Numerous examples are given of unwatered milks with less and watered milks with more than 8% of solids-not-fat. The f.p. of the genuine milks were from -0.530° to -0.550° , and of the watered milks from -0.486° to -0.512° . The f.p. is regarded as the final criterion.

J. R. NICHOLLS.

Milk from sick cows. J. KRENN (Z. Unters. Lebensm., 1928, 55, 47–49).—Analyses are given of the milk from sick cows. The values are exceptional and might suggest dilution, but the usual chemical analysis together with the f.p. and the chlorine-sugar value show these milks to be abnormal. J. R. NICHOLLS.

Effect of working of cows on the composition of the milk. F. HAUN (Z. Unters. Lebensm., 1927, 54, 337–341).—The chief alteration in the composition of the milk of cows put to work is in the fat content, which may increase by more than 1% in 24 hrs. The density of the milk and of the serum and the refraction all show lower values on days of rest between days of work. The yield of milk decreases greatly when the animal is put to work. W. J. BOYD.

Preservation of milk samples for fat determination. P. MARSCHEW (Milch. Zentr., 1928, 57, 85–88).—Varying quantities of formalin, potassium dichromate, and sublimate were added to milk, and after definite periods the fat was determined by three butyrometric methods. A saturated solution of potassium dichromate is regarded as the best preservative, and to keep 100 c.c. of milk at 15–19° for 14 days, 0.5–1.0 c.c. is required; for 1 month, 1.5 c.c.; and for milk at 19–20° for

14 days, 1.5 c.c. Using formalin, 100 c.c. of milk at 15–19° require 0.2 c.c. to keep it for 14 days and 0.3 c.c. for 1 month, and the fat must be determined by the Gerber process, using sulphuric acid of d 1.815 or above. To keep a similar quantity of milk for 14 days at 15–19°, 0.6–0.8 c.c. of 5% solution of sublimate is necessary.

J. R. NICHOLLS.

Determination of milk fat in small quantities of fat. J. GROSSFELD and F. WISSEMAN (Z. Unters. Lebensm., 1927, 54, 352–356).—For the examination of fat extracted from foodstuffs of low fat content, the ordinary methods are unsuitable, owing to the large quantity of material which has to be treated. The semi-microchemical methods of Lührig (B., 1922, 725 A) for the determination of the Reichert-Meißl and saponification values are described. For these, the amounts of sample and reagents used are reduced to one tenth of those required for the ordinary methods. The author describes a similarly reduced semi-microchemical method for the determination of the butyric acid value. The distillate of 11 c.c. is titrated with 0.01*N*-sodium hydroxide solution. The results are in agreement with those obtained by the macro-method. W. J. BOYD.

Determination of chlorine and sodium in mammalian milks. L. BARTRE and E. DUFILHO (Ann. Falsif., 1928, 21, 98–100).—Mares' milk was found to have less than 0.006 g. of sodium per litre; during the colostrum period it contained over 1 g. of chlorine per litre and later between 0.5 and 0.7 g. per litre. Human milk also contained at first over 1 g. of chlorine per litre and later between 0.6 and 0.7 g., and the sodium content was negligible till after about the 45th day. It is concluded as a result of these and previous investigations that sodium is not necessary to the newly born, and serves later for the combination of other necessary elements: mares' milk may to a certain extent be substituted for human milk, but after the fifth month of lactation the latter more closely resembles cows' milk.

D. G. HEWER.

Detection of adulterants in cacao butter in chocolate. COLOMBIER and CHAIZE (Ann. Falsif., 1928, 21, 91–97).—The following adulterants of cacao butter were divided into two classes, viz. (a) comprising "Kunérat, Chocotal, and Karamélor," and (b) "Illipé, Pontianak, and Banka," with "Kayao" in a separate class. For a sample of 100–125 g. of chocolate 10 g. are extracted for total fat, 20 g. are used for sugar determination, and the remainder is extracted with carbon tetrachloride and on the residue are determined, after two days, (1) refractive deviation, (2) soluble and (3) insoluble volatile acids, and (4) the H.M. value, derived from a modification of Halphen's method (B., 1909, 841) which consists in dissolving 1.5 g. of fat in 3 c.c. of carbon tetrachloride, adding drop by drop a solution of bromine in an equal volume of carbon tetrachloride until an orange colour is obtained, shaking, filtering, and adding to the clear filtrate an equal volume of light petroleum, when, with fats containing enough Banka or illipé butters, a white band forms at the surface of contact; a turbidity in the light petroleum is visible with 5% of these fats. If the sum of values (2) and (3) is greater than (1), a fat of class (a) is present, and if (4)

is positive a fat of class (b). If (4) is negative, and (2) and (3) less than (1) all fats of classes (a) and (b) are absent, whilst one fat of each group present together may also be detected. To detect Kayao fat the fact that a blue fluorescence forms in Wood's light is utilised. The method is applicable to nut and milk chocolates.

D. G. HEWER.

Determination of coating material in coffee. A. SCHUGOWITSCH (Z. Unters. Lebensm., 1927, 54, 330–336).—In Austria and other countries the use of coating material such as colophony or sugar for brightening coffee is controlled by defining the permissible proportions of ether, alcohol, and water extracts. It is shown that whilst these values are nearly constant for all kinds of raw coffee, in roasted coffee the alcohol- and ether-soluble portions vary according to the kind of coffee and the degree of roasting. The aqueous extract increases with increased roasting, but is nearly constant for all varieties. In very strongly roasted beans the amount of ether and alcohol extract is a multiple of that in normally roasted beans and exceeds very considerably the limits hitherto permitted. In normally roasted beans (dull brown) the ether and alcohol extract never exceeds 1% nor the aqueous extract 0.50%; if these values are exceeded artificial brightening may be assumed with certainty. During storage the ether extract of roasted coffee almost always increases and the alcohol extract decreases. For the detection of colophony in coffee, 10 g. are shaken for 3 min. with 30 c.c. of 0.5*N*-caustic soda and some kieselguhr. The filtered extract is acidified with dilute sulphuric acid and shaken with ether. The ether extract is washed with water, evaporated, and the residue dissolved in 1–1.5 c.c. of acetic anhydride. To this in an inclined test-tube a drop of sulphuric acid (62.5%) is carefully added, whereupon a blue-violet coloration develops, changing to dirty brown on shaking and finally to yellow. In the absence of colophony no colour or only a faint rose tint develops.

W. J. BOYD.

Acidity of honey and artificial honey. J. FIEBE and W. KORDATZKI (Z. Unters. Lebensm., 1928, 55, 59–63).—Forty-two samples of honey and 12 of artificial honey have been examined. The p_H values for natural honeys were about 3.9 (limits 3.8 and 4.3); and for artificial honeys 3.2 (limits 3.0 and 4.0). The number of c.c. of *N*-alkali required to neutralise 100 g. to phenolphthalein was 0.6–4.0 for pure honey and 0.38–0.90 for artificial. Mixtures gave p_H values sometimes within and sometimes outside the limits for pure honey.

J. R. NICHOLLS.

Separation of honey. J. FIEBE (Z. Unters. Lebensm., 1928, 55, 64–65).—A honey which was apparently regularly crystallised had a dazzling white surface layer sharply differentiated from the remainder. The honey was examined in three portions, viz., the surface layer, the upper half of the remainder, and the lower half. The white layer was richer in sucrose, dextrose, salts, and non-sugars. The water contents of the three layers from top to bottom were 6.6%, 15.7%, and 11.25%. It is assumed that shaking had aerated the honey, and that on being kept the air bubbles had risen, carrying sugar particles etc. to the surface. The top layer had

lost water by evaporation, and the bottom layer had less water than the middle on account of crystallisation of sugar.

J. R. NICHOLLS.

Keeping qualities of frozen meat, especially frozen sausage meat. O. ACKLIN (Z. Unters. Lebensm., 1928, 55, 31—44).—Employing appearance, smell, and bacteria ratio as criteria, no fundamental difference with regard to deterioration was observed between frozen and native meat after storage at 4°, 20°, and 37° in raw and in cooked states. No difference was observed between first-quality native sausage meat and 10—16 months' old frozen meat after storage at an average temperature of 15° and at an average humidity of 60%, with systematic control of character, oxygen consumption, methylene-blue reduction, and acidity and rancidity of the fat.

J. R. NICHOLLS.

Treatment of meats via the blood stream. A. GAUDUCHEAU (Ann. Falsif., 1928, 21, 84—90).—Flesh of chickens, rabbits, pigeons, etc. may be flavoured as required by injecting suitable substances directly into the blood stream. The muscles readily take up the aroma, and the changes taking place are being studied with a view to the commercial application of the technique.

D. G. HEWER.

Rapid test of pepper powder for excessive chalk content. W. PLAHL (Z. Unters. Lebensm., 1927, 54, 369—371).—A suitable quantity of pepper powder is placed on a microscope slide glass, quickly mixed with 2 or 3 drops of chloral hydrate solution containing about 1% of dilute hydrochloric acid, and covered with a cover glass. The evolution of bubbles of carbon dioxide is noted. Some trials with unadulterated pepper, and with pepper of known chalk content, give the experience necessary for the recognition of adulterated samples. With ash content up to 7% in black pepper, and 4% in white pepper, only very slight evolution of gas occurs, in bubbles visible under the microscope or with a strong lens. In samples with 13% or 14% of ash, the bubbles are readily seen with the unaided eye.

W. J. BOYD.

Chemical analysis of nutritive preparations. E. KOMM and R. MÜLLER (Z. Unters. Lebensm., 1928, 55, 53—59).—The analysis of such preparations must be sufficient to indicate their compositions so that conclusions can be drawn as to their biological values. An example is given of the examination of "Provita" in which the following were determined:—water, total and water-soluble sugar substances, total lipoids, cholesterol, ether-soluble and alcohol-soluble phosphatides, fat, nitrogen (total, lipid, and water-soluble), tyrosine, tryptophan, fibre, total and water-soluble ash (and its analysis).

J. R. NICHOLLS.

Technique of biological protein differentiation. A. BRÜNING and B. KRAFT (Z. Unters. Lebensm., 1927, 54, 347—352).—A semi-microchemical method for the differentiation of proteins by the precipitin reaction is described. The tubes used are 63—65 mm. long, with internal diameter 4.5 mm. The pipettes are 12 cm. long and 3.5 mm. wide with drawn-out points 12 mm. long, so adjusted in aperture that the liquid flows out in a series of drops. The tubes are placed in a stand and held firmly by their middles by metal clips

so that the liquid can be freely observed. For greater quantities of material tubes 7.5 mm. wide and 65 mm. long can be used. A simplified technique for the Hauser capillary method is also described.

W. J. BOYD.

Detection and determination of methyl *p*-hydroxybenzoate in foodstuffs. F. WEISS (Z. Unters. Lebensm., 1928, 55, 24—31).—Methyl *p*-hydroxybenzoate, recommended as a preservative for foodstuffs etc. (cf. U.S.P. 1,627,342; B., 1927, 457), appears in commerce under the names of "Solbrol" and "Nipagin." It can be extracted quantitatively from weakly acid solutions by means of ether or a mixture of light petroleum and ether, and the solvent can be evaporated without loss if the temperature is not allowed to exceed 40°. Potassium ferrocyanide and zinc acetate may be used as cleaning agents to remove proteins without carrying down any appreciable quantity of the ester. From fats and oils it can be partially extracted with hot water or by steam distillation, but in neither case is the extraction complete. With Millon's reagent the ester gives a gradually increasing red colour, the non-appearance of which within 15 min. indicates its absence. Salicylic acid under the same conditions gives a brownish-red colour which is, however, usually distinguishable. The ester does not interfere with the tests for salicylic and benzoic acids and only large quantities interfere with the test for mikrobin (B., 1927, 795). The colour produced with Millon's reagent can be compared with that produced under the same conditions from known quantities, or the methyl alcohol can be determined after hydrolysis with potash or by Zeisel's method.

J. R. NICHOLLS.

Report of the Government Laboratory at Amsterdam for 1927. J. STRAUB (Chem. Weekblad, 1928, 25, 161—165).—A brief account of the lines along which original work in connexion with the examination of milk, edible fats, cheese, eggs, flour, and bread has been conducted.

S. I. LEVY.

Composition of Camembert cheese at various stages of ripening. M. P. BABKIN (Trans. Vologda Dairy Inst., 1926, Bull. 65, 205—206).

Rancidity in butter. INIKHOV and SHOSKIN. **Cereal oils.** MUNRO and BINNINGTON.—See XII. **Vitamin-B in wheat.** HUNT.—See XVI. **Lecithins.** MATTHES and BRAUSE.—See XX.

PATENTS.

Manufacture of stable dry substances from colloidal liquids, especially liquids containing fat and albumin. C. KNOCH and F. GROSS (B.P. 285,313, 21.7.27).—After concentration of the colloidal liquid, e.g., milk, by evaporation below 60°, a part of its soluble salts and sugar content is removed by osmosis and evaporated either to dryness or to a crystallisable syrup. The product is then rapidly dried in the form of separate drops on glass or china rollers heated by steam below 60°. The granules are scraped off the rollers, compressed into shapes to remove occluded air, and coated with an air-tight layer by "glazing" with the syrup or by "dragging" with the dried powder of salts and sugar.

F. R. ENNOS.

Treatment of vegetable produce. J. B. BUTLER

and J. J. DRUMM (U.S.P. 1,666,551, 17.4.28. Appl., 7.10.26. U.K., 30.11.25).—See B.P. 266,069; B., 1927, 314.

Apparatus for producing refined cocoa liquor from cacao nibs and the like. W. E. PRESCOTT, BAKER PERKINS, LTD., and ANC. ÉTABL. A. SAVY, JEANJEAN & CIE., SOC. ANON. (B.P. 287,628, 22.12.26).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Evaluation of saponin drugs. L. KOFLER and P. A. ADAM (Arch. Pharm., 1927, 265, 624—652).—Physical and chemical methods of determining the strength of saponin drugs are faulty, and reliance has therefore necessarily been placed on determinations of hæmolytic potency. It is now shown that this method is subject to hitherto unsuspected errors: Thus the hæmolytic index of isolated saponins is influenced considerably by the hydrogen-ion concentration of the medium, and in such a way that they can be divided into two classes (cf. Kofler and Lázár, A., 1928, 318). In extracting the powdered drugs, the "reaction" of the medium therefore needs careful control. Further, in any series of tests, only one sample of blood may be used, and from that the corpuscles must be preferably unwashed, as the presence of serum has the same effect on the hæmolytic index of drug extracts as on that of a pure saponin (cf. Kofler and Lázár, *loc. cit.*). Earlier published tables of the resistances of different kinds of blood corpuscles (cow, man, rat, etc.) to saponins are quite untrustworthy, as the order of resistances depends on the particular saponin, or drug, under investigation. Differences are seen in the resistances of blood samples from different individuals of the same species. Values for the hæmolytic index (the authors adhere to Kobert's method of indicating strengths) are thus only comparable among themselves for one series of tests, unless a standard saponin is used as a control. For this purpose "saponin pur. albiss." (Merck) is used; this has a hæmolytic index, towards unwashed cow's blood corpuscles, at p_H 7.4, of about 25,000. It should be used in freshly prepared solution. The extraction of the drugs is facilitated by using an alkaline medium, but the alkalinity must be controlled. A phosphate buffer of p_H 7.4, $M/30$, in 0.9% sodium chloride solution is therefore used. This gives directly a solution suitable for hæmolytic tests. The extraction is carried out at water-bath temperature for 30 min. (0.25 g. of drug to 50 c.c. of solution). When the mixture is boiled irregular results are obtained, the saponins apparently undergoing change. When extraction is carried out at room temperature it is incomplete even after 6 hrs. Sarsaparilla, however, behaves abnormally when heated and must be extracted in the cold. The drugs are not satisfactorily extracted in the form of chips, and must be ground, but not powdered, to pass a "No. 5" sieve. Methyl or ethyl alcohol as an extracting medium is unsatisfactory and less efficient than a mildly alkaline saline solution, and itself interferes with the hæmolysis. The suspension of blood corpuscles is conveniently 2—5%, and is made up from defibrinated blood with the phosphate buffer saline solution specified. It is found that the lethal strength of extracts of saponin drugs

towards tadpoles runs parallel with the hæmolytic index, and this property may also be used in determining the strength of such extracts. Many samples of various drugs were examined. Radix senegæ has a hæmolytic index of about 4000 and is fairly constant in strength; radix saponariæ albæ is similar, with an index of about 10,000; cortex quillaia, radix primulæ, and radix saponariæ rubræ show lower and less constant values, as also does herba herniariæ; radix sarsaparillæ varies over a wide range (hæmolytic index from below 100 to above 3000) and to this variation is attributed the discrepant statements regarding the value of the drug in pharmacy.

W. A. SILVESTER.

Preparation of decoctions of saponin drugs. L. KOFLER and P. A. ADAM (Arch. Pharm., 1927, 265, 652—653).—That the saponins are more thoroughly extracted from the drugs when these are ground rather than in the form of chips has been demonstrated in the preparation of solutions for analysis (cf. preceding abstract). It is now shown that commercial decoctions are also preferably made from ground drugs. The powder should pass a "No. 5" sieve. Frequently, *e.g.*, with water alone, more than twice the usual amount of saponin is extracted. The addition of a small amount of sodium carbonate is beneficial. Extraction may be carried out at the b.p. except with sarsaparilla, which must be extracted cold.

W. A. SILVESTER.

Action of picric acid on tropine silicotungstate. R. HAZARD (J. Pharm. Chim., 1928, [viii], 7, 255—262). Tropine solutions which are too dilute or too acid to give a precipitate with 10% silicotungstic acid solution do so when treated with aqueous picric acid solution, the amount and speed of precipitation being proportional to the amount of tropine in solution. The yellow precipitate is a silicotungstate, and it may be freed completely from both the yellow colour and picric acid by washing with hydrochloric acid. Atropine silicotungstate is very sparingly soluble, and does not give this reaction, which affords a method of separating and identifying the two bases, in which case it is advantageous to work in 2% hydrochloric acid. The hydrolysis of atropine may be followed in this way. ψ -Pelletterine gives a similar precipitate after treatment with silicotungstic acid followed by picric acid in 2% hydrochloric acid.

S. COFFEY.

Determination of morphine in opium. V. MACRÌ (Boll. Chim. Farm., 1928, 67, 129—131).—Eder and Märki's method (Pharm. Acta Helv., 1927, [2—4]) is discussed. The use of benzene in place of ether for the extraction of the opium does not seem to be of any great advantage, and the author prefers to employ a saturated aqueous solution of morphine for this purpose, and to effect only the final washing with ether or benzene. The introduction of extraneous substances such as sodium carbonate or borax involves possible error and is to be avoided.

T. H. POPE.

Cocaine and Maclagan's test. P. BRETEAU (J. Pharm. Chim., 1928, [viii], 7, 329—331).—The variations of Maclagan's test for isotropylcocaine (truxilline) in different pharmacopœias are discussed and the following procedure is recommended: 0.1 g. of the cocaine salt is dissolved in 80 c.c. of water, and 2 c.c. of 1% ammonia

solution ($d 0.995$) are added, stirring with a wooden or rubber-covered rod. After settling for 15 min. the liquid should be clear. The sides of the vessel are then rubbed from time to time, but not too vigorously, with a glass rod. A crystalline deposit of cocaine should separate and the supernatant liquid should be clear. The presence of isotropylecocaine is indicated by the formation of an opalescent liquid which is cleared by the addition of hydrochloric acid. E. H. SHARPLES.

Fructus papaveris and sirupus papaveris as possible poisons. L. VAN ITALLIE and A. J. STEENHAUER (Arch. Pharm., 1927, 265, 698—705).—Poisoning has been known to result from the administration of sirupus papaveris, which is a popular soporific for infants. There are considerable discrepancies in published figures for the percentage of morphine in both the fruit and the extract. It is now found that much more morphine may be present than is commonly supposed. Thus *Fructus papaveris* contains 0.4—0.5% of morphine, all of which passes into an aqueous extract; the syrup may contain 0.01—0.06%. In detecting morphine, Pellagri's reaction is modified: an excess of iodine is used, and the excess is removed by addition of sodium thiosulphate; 0.05 mg. of morphine can then be detected. In determinations the procedures of Georges and Gascard (B., 1906, 779) and Mai and Rath (B., 1908, 828) give almost identical results. In toxicological examinations the extract obtained by the Stas-Otto method should be purified. Other methods are compared. W. A. SILVESTER.

Composition of commercial plant lecithin. H. MATTHES and G. BRAUSE (Arch. Pharm., 1927, 265, 711—712).—Plant lecithins have a higher iodine value (90—100) than egg lecithin. The phosphorus:nitrogen ratio in egg lecithin is always less than 2.21 (%: %), whereas in plant lecithin it is greater than 2.21, i.e., there is more, instead of less, phosphorus than corresponds with the theoretical ratio of 1 atom of each element. Five commercial plant lecithins showed moisture varying from 1.13 to 4.92%; phosphorus from 2.30 to 3.45%; nitrogen from 0.72 to 1.51%; phytosterol from 0.65 to 1.71%; iodine value from 90.0 to 100.2; acid value from 26 to 123.

W. A. SILVESTER.

Differentiation of animal and plant lecithins. H. MATTHES and G. BRAUSE (Arch. Pharm., 1927, 265, 708—710).—Commercial animal lecithins have been found (Brause, Diss., Königsberg i. Pr., 1926) to contain 0.22—3.38% of cholesterol, although Cohn (Z. öffentl. Chem., 1913, 59) has stated that this compound should be absent. Even material prepared in the laboratory contains 0.44%, and so it is evident that all lecithins of animal origin can be presumed to contain cholesterol. Plant lecithin is likewise found to be impure, and to contain, according to its commercial source, from 0.70 to 1.75% of phytosterol. This provides a means of determining whether any particular lecithin is of plant or animal origin, or is a mixture. The cholesterol, or phytosterol, is converted, through the digitonide, into the acetate, using the methods of Windaus and Fahrion (cf. Grün, "Analyse der Fette und Wachse," 1925, 1, 206, 265). Cholesteryl acetate from egg lecithin has, in the

crude state, m.p. 112.5—113.8°, and, after recrystallisation from absolute alcohol, m.p. 115°. Phytosteryl acetate similarly has m.p. 129—130° (crude) or 131.5—133.5° (recrystallised). A mixture of lecithins gives a steryl acetate the m.p. of which lies between these limits and is not constant on repeated crystallisation.

W. A. SILVESTER.

State of dispersion of [commercial] preparations of colloidal silver. H. BECHHOLD and E. HEYMANN (Arch. Pharm., 1927, 265, 669—675).—"Collargol" (the original preparation), four other German preparations, and three other samples, of French, Swiss, and Polish origin, respectively, have now been examined. All, except the Polish preparation, which contains 75%, have the normal proportions (about 25%) of protective colloid. Wide differences are, however, shown in the degree of dispersion of the silver. The samples were analysed before and after being centrifuged, to give the proportion of relatively large particles, filtered through an ultra-filter, to give the proportion of very small particles and ionic silver, and examined ultramicroscopically to give the mean size of the particles. They were also kept for 8 months and the precipitate, if any, was examined. "Collargol" contains 74% Ag; it gives a very small precipitate after 8 months; the mean size of the particles is 19 $\mu\mu$ (they are assumed to be cubes, and the length given is that of the edge); no separation takes place when a 1% solution is centrifuged, but a fair proportion passes the ultra-filter. The other samples contain particles the mean size of which varies from 19 $\mu\mu$ to 32 $\mu\mu$ but in some samples, although the mean size of the particles may be low, yet they are far from being uniform. The worst sample, viz., the Polish preparation, contained particles of mean size 35 $\mu\mu$, and gave separations both on centrifuging and keeping, although so heavily loaded with protective colloid.

W. A. SILVESTER.

Rate of dissolution of essential principles during percolation. I. Press cake from bitter almonds. M. BRIDEL and (Mlle.) M. DESMAREST (J. Pharm. Chim., 1928, [viii], 7, 153—162, 201—207; cf. A., 1928, 201).—The press cake from bitter almonds containing both invertase and β -glucosidase furnished 1.73% of reducing sugar, 3.47% of sucrose, and 6.84% of amygdaloside on repeated extraction with boiling 70% alcohol, whilst percolation in the cold with one quarter of the amount of alcohol afforded 1.76% of reducing sugar, 3.65% of sucrose, and 7.35% of amygdaloside. The sucrose is very rapidly hydrolysed during maceration, but amygdaloside is more resistant, being decomposed to the extent of 10% in 10 days. A study of the rates of dissolution proved that all the above constituents are completely extracted from press cake by percolation with five times its weight of 70% alcohol, the sucrose being removed most rapidly and amygdaloside least rapidly. The sucrose is extracted in 5 hrs., and the amygdaloside in 12 hrs., this rapid extraction being the reason why the sugar is not hydrolysed by the invertase present. The sugars and glucoside together account for rather more than 50% of the total extracted solids. Pure amygdaloside is obtained in 3% yield by percolation with 70% alcohol, and the residual cake on maceration with water furnishes 1.4% of emulsin as a reddish

powder. The yield of emulsin from the press cake is only one half that from the corresponding quantity of almonds. A comparison of the enzyme with that from almonds shows that the latter is the more active with concentrated aqueous solutions of dextrose, but no difference is observed in methyl alcoholic solution. Emulsin from the press cake hydrolyses salicoside and lactose more rapidly than that from almonds, but no difference is found with galactose and sucrose. S. COFFEY.

Terpineol from terpin. A. ROBERT (Bull. Inst. Pin, 1927, 153—156, 177—182; Chem. Zentr., 1927, ii, 2295).—Dehydration of terpin hydrate by boiling with a little phosphoric or sulphuric acid yields, in the first runnings, dipentene, terpineols, α - and γ -terpinene, and cineole, but chiefly α -terpineol. Details are given for the detection of these substances.

A. A. ELDRIDGE.

Insecticides. FRANÇOIS and SEGUIN.—See XVI. PATENTS.

Manufacture of ammonium sulphoichthyolate. D. H. ROWLAND, Assr. to AMER. ICHTHYOL OIL CO. (U.S.P. 1,664,376, 27.3.28. Appl., 4.2.27).—Petroleum is sulphonated and heated while being agitated. The heating is stopped, the agitation continued, and the mixture diluted with kerosene. A salting-out reagent is added, the whole agitated until frothing ceases, and the ammonium sulphoichthyolate isolated by adding ammonia solution until the mixture is alkaline and again diluting with kerosene. F. G. CLARKE.

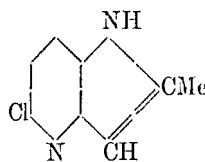
Diallylcyanamide. E. B. VLIET, Assr. to ABBOTT LABORATORIES (U.S.P. 1,659,793, 21.2.28. Appl., 12.4.26).—An allyl halide is heated with disodium cyanamide in aqueous-alcoholic solution to yield *diallylcyanamide*, b.p. 105—110°/18 mm. and 140—145°/90 mm., which is readily hydrolysed to diallylamine.

T. S. WHEELER.

Manufacture of salts of diethylaminoethyl *p*-aminobenzoate. F. HOFFMANN-LA ROCHE & Co. A.-G. (Swiss P. 118,336, 17.10.25, and addn. Swiss P. 120,447, 17.10.25).—An equimolecular mixture of β -diethylaminoethyl *p*-aminobenzoate and glutamic acid in aqueous solution is evaporated to dryness in a vacuum, or the ester hydrochloride and sodium glutamate may be used, the dry residue being extracted with alcohol and the alcohol removed in a vacuum. An alcoholic solution of *p*-aminobenzoic acid and β -diethylaminoethyl *p*-aminobenzoate deposits non-hygroscopic crystals of the salt, m.p. 104°. These salts have stronger anaesthetic power than the ester hydrochloride. C. HOLLINS.

Production of new pyridine derivatives. DEUTS. GOLD- & SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 259,982, 14.10.26. Austr., 17.10.25).—3-Pyridylhydrazine, or a derivative of 2- or 3-pyridylhydrazine, is condensed with aldehydes or ketones, keto-esters, etc., and the products in suitable cases are converted into pyrazolones or indoles. *Benzaldehyde-3-pyridylhydrazine*, m.p. 165—166°, *ethyl β -6-chloro-3-pyridylhydrazinocrotonate* (from ethyl acetoacetate), m.p. 123—124°, *6-iodo-3-pyridylhydrazine* (from 6-chloro-3-iodopyridine and hydrazine hydrate), m.p. 124°, *ethyl β -5-nitro-2-pyridylhydrazinocrotonate*, m.p. 93—95°, *5-nitro-2-pyridylhydrazones of acetone*, m.p. 168°, *propaldehyde*, m.p. 151°, and

benzaldehyde, m.p. 226—228°, are described. By heating the compound from 5-nitro-2-pyridylhydrazine and ethyl acetoacetate at 100° there is formed 1-(5-nitro-2-pyridyl)-3-methyl-5-pyrazolone, m.p. 240—260°. Acetone 5-nitro-2-pyridylhydrazine, heated with zinc chloride at 200°, gives 5-chloro-2-methyl- β -pyrindole (annexed formula), m.p. 208—210°; 5-chloro-3-methyl- β -pyrindole, m.p. 199—202°, is similarly obtained from the propaldehyde compound.



C. HOLLINS.

Manufacture of derivatives of 2-aminopyridine. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 265,167, 6.1.27. Ger., 27.1.26).—Alkylated 2-aminopyridines having more than two carbon atoms in the *N*-substituent group or groups are obtained either from 2-chloropyridine and an aliphatic amine, or from sodio-2-aminopyridine and an aliphatic halide. 2-Diethylaminopyridine, b.p. 208—214°, 2-isopropylaminopyridine, b.p. 105°/16 mm., 2-allylaminopyridine, b.p. 124—129°/18 mm., 2-isoamylaminopyridine, b.p. 135—140°/12 mm., and 2-cetylaminopyridine, m.p. 65—66°, 215—225°/2 mm., are described. C. HOLLINS.

Manufacture of barbituric acid soporifics. O. Y. IMRAY, From I. G. FARBERIND. A.-G. (B.P. 285,598, 7.12.26).—Barbituric acid derivatives containing an acetylenic linking in one or both of the 5-substituent groups are good hypnotics producing no bad after-effects. 5-Propargyl-5-isopropylbarbituric acid, m.p. 165°, is obtained from 5-isopropylbarbituric acid, alcoholic sodium hydroxide, and propargyl bromide. Butinen- γ -ol (Hess and Munderloh, A., 1918, i, 291) gives with hydrogen bromide γ -bromobutene, from which 5- γ -methylpropargyl-5-isopropylbarbituric acid is similarly obtained. By the action of the magnesyl compound of isopropenylacetylene (γ -methylenebutene), the dehydration product from γ -methylbutene- γ -ol (*loc. cit.*), on formaldehyde there is produced α -isopropenylpropargyl alcohol, $\text{C}_6\text{H}_5 : \text{CMe} : \text{C} : \text{C} : \text{CH}_2 : \text{OH}$, b.p. 68—69°/11 mm., which is converted by phosphorus tribromide into α -isopropenylpropargyl bromide, b.p. 65—67°/20 mm.; from this is prepared 5- α -isopropenylpropargyl-5-isopropylbarbituric acid, m.p. 157°. 5-Propargyl-5-sym.-sec.-amylbarbituric acid, m.p. 175—178°, is obtained from propargyl bromide and the barbituric acid prepared from ethyl sym.-sec.-amylmalonate, b.p. 114—116°/9 mm.

C. HOLLINS.

Manufacture of complex gold-nucleic acid compounds. SOC. CHEM. IND. IN BASLE (GES. F. CHEM. IND. IN BASEL) (B.P. 276,677, 25.8.27. Switz., 25.8.26).—Complex compounds (insoluble in acid), in which the gold cannot be detected by ordinary reagents, are formed by the interaction of nucleic acids or their soluble salts and gold compounds. B. FULLMAN.

Production of liquids of medicinal value. A. T. PORTER (U.S.P. 1,660,655, 28.2.28. Appl., 10.2.21. Renewed 25.5.27).—Hydrocarbon oils are atomised and partially burned with a limited supply of air, and the vapours obtained are condensed. T. S. WHEELER.

Vanillin and isovanillin (B.P. 285,156 and 285,551). **isoEugenol** (B.P. 271,819).—See III. **Rubber emulsions** (B.P. 286,527).—See XIV.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENTS.

Photographic developers. I. G. FARBERNIND, A.-G. (B.P. 280,525, 31.10.27. Ger., 13.11.26).—A cyanide of a heavy metal (mercury, or less effectively cobalt or cadmium) is added to any photographic developer. Mercuric cyanide (0.3 g. per litre) acts as a desensitiser, enabling development to be completed in bright yellow light; it also prevents fogging in prolonged development with weak developers. The cyanides give no precipitate with any of the usual developers. C. HOLLINS.

Mercurial printing surface. A. R. TRIST (U.S.P. 1,665,000, 3.4.28. Appl., 5.10.26. U.K., 5.11.25).—See B.P. 265,294; B., 1927, 429.

XXII.—EXPLOSIVES; MATCHES.

Season-cracking of cartridge cases. GRIMSTON.—See X.

PATENTS.

Nitration process. J. MARSHALL and F. H. BERGEIM, ASSRS. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,660,651, 28.2.28. Appl., 31.12.26).—Solid polyhydric alcohols dissolved in sulphuric acid are added to nitric acid to form the corresponding nitrates.

T. S. WHEELER.

XXIII.—SANITATION; WATER PURIFICATION.

Fumigation with hydrogen cyanide. P. BUTTENBERG and G. GAHRTZ (Z. Unters. Lebensm., 1927, 54, 376—387).—An account is given of the progress in the use of hydrogen cyanide for the destruction of pests. The advantages of using this agent in powder form (adsorbed on kieselguhr) over the tank process (in which sodium cyanide is thrown into vessels charged with dilute sulphuric acid) are discussed. Between 1923 and 1927 the former entirely displaced the tank process in the fumigation of ships in Hamburg harbour. A pungent-smelling substance is incorporated in the powder, viz., methyl bromoacetate in Zyklon B, and chloropicrin in Zyklon C. In the gaseous concentration used (0.10—3 vol.-%) hydrocyanic acid is neither inflammable nor explosive. These risks begin at a content of 15 vol.-%. Other methods of application are described. The uses mentioned include the destruction of rats and mice in ships, storehouses, and refrigerators, of insect pests in greenhouses, and of mites and insects in foodstuffs, the fumigation of furniture, cushions, fabrics, straw, asbestos, and books. Information is given concerning the concentration of the gas and time of exposure required for the destruction of pests in various materials. The retention of hydrogen cyanide by the articles exposed to it depends on such factors as the nature of the material, concentration of the gas, time of exposure, and temperature and humidity conditions during treatment. Vacuum treatment is usually effective in removing most of the retained hydrogen cyanide. For some products, such as coffee, the objection has been raised that the treatment spoils the flavour. Other commodities such as eggs for

hatching, photographic plates, etc. are rendered useless thereby. W. J. BOYD.

Determination of dissolved oxygen in water. V. G. ANDERSON and J. R. DICKSON (Chem. Eng. Min. Rev., 1927, 19, 467—468).—A modification of the Miller-Linosier method for determining dissolved oxygen in water is described. CHEMICAL ABSTRACTS.

Colorimetric determination of iron in water. P. LEHMANN and A. REUSS (Z. Unters. Lebensm., 1927, 54, 374—376).—100 c.c. of water are evaporated to dryness in a platinum basin, 5 c.c. of a 10% potassium sodium carbonate solution are added to the residue, and the contents of the basin again brought to dryness. After drying at 170—200°, the residue is heated to dull redness. When cool it is dissolved in 5 c.c. of 15% hydrochloric acid, and the solution transferred to a colourless glass cylinder with flat bottom. To the cooled clear solution, 1 c.c. of a bromide-bromate solution is added, the mixture kept for 5—10 min., and treated with 5 c.c. of a 7.5% ammonium thiocyanate solution. Simultaneously a blank test is carried out, to which a solution of iron ammonium alum of known strength is added from a burette until the colours of the two solutions are equal, both being brought to the same volume by addition of water. The amount of iron in the sample is calculated from the volume of standard iron solution added to the blank.

W. J. BOYD.

Boiler scale. WALDE.—See I.

PATENTS.

Production of disinfecting agents. A. BOEHRINGER (C. J. BOEHRINGER SOHN) (B.P. 272,543, 16.6.26. Ger., 8.6.26. Addn. to B.P. 253,918; B., 1927, 894).—Tartaric, citric, oxalic, or malic acid is used as disinfecting agent instead of, or in addition to, lactic acid, alkali being added until the pH value of the solution is 3.0 or below. Heavy-metal salts, phenols, or colouring matters which are disinfectant or antiseptic may be added, and the material may be converted into solid or semi-solid form by the addition of gelatin, with or without glycerol. W. G. CAREY.

Disinfecting and insecticidal agent. H. GÜNZLER and O. NEUBERT, ASSRS. to WINTHROP CHEM. Co., INC. (U.S.P. 1,663,883, 27.3.28. Appl., 31.3.27. Ger., 13.4.26).—A composition comprises a water-soluble salt of an aromatic aminosulphonic acid, e.g., sodium sulphanilate, and a water-soluble mercury salt, e.g., mercuric chloride. An inert wetting agent and an agglomerant miscible with water may also be present.

F. G. CLARKE.

Purification of water and removal and prevention of scale incrustations in boilers etc. G. S. NEELEY and G. WATKINS (B.P. 287,196, 12.11.26).—Feed water is subjected to the influence of an externally applied alternating electric current before being discharged from the inlet pipe, whilst an alternating current differing in voltage and phase from the former is passed through the shell and water content of the boiler.

J. S. G. THOMAS.

Treatment of sludge (B.P. 286,855).—See I.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 8, 1928.

I.—GENERAL; PLANT; MACHINERY.

Centrifugal mixers. E. BUNTZ (Chem. Fabr., 1928, 211—213).—Several types of mixers are described, the essential feature being that the two liquids, or the liquid and solid, to be mixed are fed continuously on to a rapidly rotating dish, over the edge of which the finished mixture passes. Provision is made for heating the dish and inlet pipe, for removal of gases evolved, etc. as necessary. Their use is suggested for the acid treatment of crude oils, the salting out of soaps, and the refining of fats. In some cases spiral baffles are provided. Another type of apparatus has an upper rotating plate and a lower funnel-shaped dish with common axes revolving in opposite directions. This is intended for the mixing of colours, moistening of powders, etc. C. IRWIN.

Instrument to record the carbon dioxide content of a gaseous mixture. R. GORDON and J. F. LEHMANN (J. Sci. Instr., 1928, 5, 123—126).—An instrument is described the action of which depends on the variation in electrical conductivity of a solution of barium bicarbonate in equilibrium with a saturated solution of barium carbonate. The readings are independent of the other constituents of the gaseous mixture, which is passed through the solution. C. W. GIBBY.

Value of aluminium and its alloys in chemical and allied industries. H. BUSCHLINGER (Chem. Fabr., 1928, 209—211).—The corrosion of aluminium, as of other metals, is affected by crystalline structure and surface quality, the absence of inclusions (e.g., Al_2Fe), and particularly by the quality of the water used if aqueous solutions are being handled. The possibility of the application of aluminium to plant used in tar distillation is discussed. The penetration of the protective surface layer of oxide is often the decisive factor in starting corrosion. Large aluminium vessels should on structural grounds be cylindrical rather than hemispherical. The rapid diminution in strength with rise of temperature is pointed out. Whilst the walls of vessels to withstand pressure are constructed on a theoretical basis, the minor parts are usually designed according to empirical rules. In jacketed vessels the steam inlet must be designed to minimise mechanical wear. In all cases corners and angles should be avoided to facilitate cleaning. In the case of aluminium vessels which are to contain edible preparations, rollers etc. should be used exclusively for aluminium, and the sheets should be pressed so as to give a smooth surface capable of polishing both inside and out. Suitable packing material, quite free from acid, should be used.

C. IRWIN.

Strength of curved walls exposed to external pressure. C. A. ANDSTEN (Ind. Eng. Chem., 1928, 20, 364—366).—Equations derived from experimental work

by Bach on the collapsing pressures of spheres and cylinders under external pressure are plotted graphically. A separate curve is obtained for each metal which may be used, and by using Bach's constants the necessary data are obtained by which the ratio of thickness to diameter is correlated with the pressure to be used.

C. IRWIN.

Gas cleaners. HARBORD.—See X. **Glass extractor.** FREY and REED.—See XV. **Flammability of refrigerants.** JONES.—See XXII.

PATENTS.

Rotary furnaces. GEWERKSCHAFT SACHTLEBEN, and J. KÜPPERS (B.P. 286,590, 30.9.27. Ger., 5.3.27. Addn. to B.P. 268,308; B., 1927, 927).—The inner lining of the muffle furnace consists of fireproof material of high thermal conductivity, whilst that of the outer muffle is of low conductivity.

H. ROYAL-DAWSON.

Apparatus for carrying out exothermic catalytic gas reactions. H. HARTE (B.P. 275,983, 8.8.27. Ger., 10.8.26).—The catalytic tube is divided transversely into a number of separate chambers, through each of which a corresponding fraction of the gas current passes from a common conduit, and the reaction products and uncombined gases are withdrawn through a common collecting conduit. The reaction is thus distributed as uniformly as possible over the whole catalyst and local heating is avoided. The collecting conduit may surround the contact chambers in the form of a cylinder, tube, cooler, or heat exchanger.

A. B. MANNING.

Temperature-regulating system. C. E. ANDREWS, Assr. to SELDEN Co. (U.S.P. 1,666,251, 17.4.28. Appl., 19.2.27).—The h.p. of a two-phase, liquid-vapour, temperature-regulating system which includes reflux condensation of the vapour, and in which the liquid contains a number of components of which at least one is normally solid, is regulated by varying the composition of the liquid condensed in the reflux apparatus before it is returned to the main body of the liquid.

L. A. COLES.

Production of emulsions. C. A. AGTHE (U.S.P. 1,668,136, 1.5.28. Appl., 12.3.23. Ger., 13.3.22).—See B.P. 210,307; B., 1924, 239.

Apparatus for comparing the colour of liquids by reflected and transmitted light. H. A. ELLIS, Assr. to BRIT. DRUG HOUSES, LTD. (U.S.P. 1,667,249, 24.4.28. Appl., 25.2.25. U.K., 12.11.24).—See B.P. 235,458; B., 1925, 616.

Measuring the density of gas. G. KÖNIG (U.S.P. 1,664,752, 3.4.28. Appl., 3.7.24. Ger., 6.3.20).—See B.P. 159,845; B., 1922, 692 A.

Friction surfaces (B.P. 264,471).—See X. **Gas-testing apparatus** (B.P. 288,908).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Coke for crucible steel melting. R. V. WHEELER (Fuel, 1928, 7, 148—151).—The preference shown by crucible steel workers for beehive over by-product coke is due to the readiness with which the former can be broken into pieces of suitable size without undue formation of breeze, and the fact that it burns uniformly at an economical rate and that the clinker formed does not stick to the furnace lining. These advantages arise from the absence of cross-fractures and the uniform cell-structure in beehive coke, combined with possession of the requisite "combustibility value." A special coke produced in a by-product oven from a blend of coking slack and anthracite duff behaved satisfactorily in trial melts as regards manner of burning, but was difficult to break into suitable pieces and gave rather a high proportion of breeze. A. B. MANNING.

Reactivity of coke. J. H. JONES, J. G. KING, and F. S. SINNATT (Iron and Steel Inst., May, 1928. Advance copy. 20 pp.; cf. B., 1928, 146).—A comparison of the reactivities of a number of metallurgical cokes has shown that the cokes from Yorkshire coals are distinct from those of South Wales or Durham coals, and possess a higher reactivity. The reactivity curves for the South Wales and Durham cokes are similar, the latter, however, giving slightly higher average values than the former. The differences are probably due to differences in the character of the coals used, and there is some indication that the ash is an important factor. Attempts to correlate reactivity values of metallurgical cokes with the results of "shatter" tests have met with no success. A. B. MANNING.

Reactivity of coke and a new method of determining it. R. A. DENG (Polytechn. Weekblad, 1926, 20, 246; Fuel, 1928, 7, 152—154).—Three cokes were prepared in the laboratory by carbonising an anthracite, a coking coal, and a gas coal, respectively, at 950°. Their relative "reactivities" were determined by passing air at a constant rate (5 litres/hr.) over 0.2 g. of the coke, of uniform size, placed in a boat in the tube of a furnace maintained at a constant temperature, and measuring the amount of carbon dioxide produced. This was done by observing the variation in electrical conductivity of a solution of barium hydroxide through which the issuing gas was passed. The coke from the anthracite had the least reactivity, and that from the gas coal the greatest. The distinction between "reactivity" and "combustibility" is emphasised. A. B. MANNING.

Specific heats of amorphous carbon and semi-cokes. E. TERRES and H. BIEDERBECK (Gas- u. Wasserfach, 1928, 71, 265—268, 297—303, 320—325, 338—345).—Three samples of amorphous carbon have been prepared, (a) by the action of sodium on barium carbonate at 550°, (b) by the action of chlorine on turpentine below 600°, and (c) by the catalytic decomposition of methane below 600°, and determinations made of the mean sp. heat of each sample, over temperature ranges from 20—300° to 20—1200°. The method used is described in detail (cf. Terres and Schaller, B., 1923, 214 A), all the precautions being taken necessary to ensure accuracy. The results for all three samples of

carbon were similar; the mean sp. heat rose rapidly with the temperature (*i.e.*, the upper temperature of the range) up to about 600°, and thence only slightly to about 1150°, the value then agreeing with that of graphite. The values obtained at the lower temperatures were at first greater than the corresponding values for graphite, but after the carbon had been heated to 1200° the mean sp. heats re-determined at the lower temperatures approximated closely over the entire range to those of graphite. Semi-cokes were prepared from four coals at temperatures from 400° to 900°, and their sp. heats have been determined over ranges up to their respective temperatures of carbonisation. The sp. heat of each coke rises with the temperature, and for cokes from the same coal is higher the lower the temperature of carbonisation. The sp. heat of the coke-substance of all the semi-cokes investigated over temperature ranges from 550° to 900° was constant and equal to 0.3825 ± 0.0025 . Below 550° the sp. heat is higher for semi-cokes from gas coals than from other coals.

A. B. MANNING.

Explosions in closed vessels. Correlation of pressure development with flame movement. O. C. DE C. ELLIS and R. V. WHEELER (Fuel, 1928, 7, 169—178; cf. B., 1925, 388).—By recording on the same film the movement of the flame produced when mixtures of carbon monoxide and air are ignited centrally in a spherical vessel and the reading of the pressure gauge, it has been shown that, except for mixtures in which the flame front travels very slowly, the moment of maximum pressure coincides with the arrival of the flame front at the boundary of the vessel within less than half a millisecond. Similar records of explosions within a cubical vessel show that pressure continues to be developed slowly after the flame front has touched the walls of the cube and while it is squeezing itself into the corners. The moment of attainment of maximum pressure does not synchronise exactly with the moment of "complete inflammation" of the mixture, but is antecedent to it, probably owing to cooling of the gases at the faces of the cube before the flame front has arrived at the corners.

A. B. MANNING.

Limits of inflammability of gases and vapours. H. F. COWARD and G. W. JONES (U.S. Bur. Mines Bull. 279, 1928. 99 pp.).—All the available data in the literature relating to the limits of inflammability of mixtures of gases and vapours with air, oxygen, or other "atmospheres," and to the variation of the limits with the initial conditions of pressure, temperature, turbulence, humidity, etc., have been collected and co-ordinated.

A. B. MANNING.

Use of active charcoal in the gas industry. A. ENGELHARDT (Gas- u. Wasserfach, 1928, 71, 290—297; cf. B., 1922, 659 A).—The properties of active charcoal are discussed in relation to its use as an adsorbent or as a catalyst in the gas industry. Methods for the determination of benzol in coal gas, and details of the large-scale plant for benzol recovery by absorption in active charcoal, are described. The benzol is now recovered from the charcoal and the latter regenerated by the use of wet instead of superheated steam, the charcoal being maintained at 100—120° by means of a heater within

the filter. Loss of material due to dust formation is small; some loss of activity occurs due to the absorption of unsaturated hydrocarbons which polymerise, depositing resinous substances within the pores of the charcoal. Hydrogen sulphide is removed from coal-gas or water-gas by catalytic oxidation in the presence of active charcoal. Small quantities of air (3–4%) and ammonia (0.3 g./m.³) are added to the gas, which is then passed through a charcoal filter. The sulphur formed is adsorbed by the charcoal, which is regenerated by extraction with ammonium sulphide followed by steaming. The polysulphide solution obtained is decomposed by heat and the sulphur recovered in a pure state. Details of the necessary plant are briefly described.

A. B. MANNING.

Coke-oven and gas-works gas: a comparative study of gas quality. R. NÜBLING and R. MEZGER (Gas-u. Wasserfach, 1928, 71, 304–310).—The importance of establishing suitable standards of gas quality is emphasised, particularly in view of the recent developments in long-distance transmission. The standards of calorific value, density, sulphur content, etc. of the gas supplied to which the gas and coke-oven industries respectively conform are compared in detail. Those of the coke-oven industry are not sufficiently rigid to satisfy general industrial demands. A. B. MANNING.

Application of low-temperature carbonisation to gas-producer practice. S. UCHIDA (Fuel, 1928, 7, 179–185).—The results obtained by the carbonisation of Japanese coals, lignite, slurry, and an oil shale in a specially designed producer are summarised; tar and gas yields and analyses etc. are tabulated, and carbon, hydrogen, and thermal balances calculated. The plant has a capacity of 3 tons/24 hrs., and consists of a producer proper surmounted by a retort, in which the coal undergoes a preliminary carbonisation at about 500°. The plant operates smoothly provided that the coal used is not too strongly swelling. The tar yield from Bibai coal (moisture 3.74%, volatile matter 42.74%, “fixed carbon” 45.07%, ash 8.45%) was 10.6%, corresponding with 81.6% of that produced in the Fischer assay.

A. B. MANNING.

K.S.G. low-temperature process. J. N. HAZELDON (Fuel, 1928, 7, 155–160).—The low-temperature carbonisation plant in operation near Essen is described. The retort comprises two concentric mild steel drums, the outer 72 ft. long and 9 ft. 9 in. in diam., the inner 84 ft. long and 5 ft. 7 in. in diam., which are inclined at a slight angle to the horizontal and are supported on rollers; in operation they are rotated at $\frac{3}{4}$ r.p.m. Fine coal is fed continuously into the lower end of the inner drum and is carried by helical flanges to the upper end; it passes then into the annular space between the drums and, under the combined effect of gravity and the rotation, returns to the lower end and is discharged as coke. Carbonisation is effected by a combination of external and internal heating. The retorts are enclosed in heating chambers through which circulate hot gases from a combustion chamber below, and superheated steam is admitted into the annular space between the drums in such a way that it passes through

the mass of the coal. The gases and vapours evolved pass through a dust catcher to a condensing system. After separation of the fines, which may amount to 20% and are utilised for blending purposes, the lump coke forms a suitable domestic fuel. It contains 10–12% of volatile matter. A Welsh coal (moisture 3%, volatile matter 32.5%, ash 5%) yielded 15 cwt. of coke, 16.1 gals. of tar, 3500 cub. ft. of gas (850 B.Th.U./cub. ft.), and 2 gals. of light spirit. The heat required for carbonisation of 1 lb. of coal is about 700 B.Th.U.

A. B. MANNING.

Composition of tar from low-temperature carbonisation of Utah coal. I. R. L. BROWN and B. F. BRANTING (Ind. Eng. Chem., 1928, 20, 392–396).—Utah coal distilled in a continuous vertical retort with superheated steam at a maximum temperature of 700° yielded 9.6% of solid tar with air condensation and a further 3.9% of fluid tar in a water condenser. The aqueous condensate was neutral to litmus, but contained small quantities of tar acids (including phenol and the cresols), ammonia, pyridine bases, and carboxylic acids. The fluid tar (d_{20}^4 0.9337) contained 23.4% of phenols, 66% of neutral oils, 1.7% of crude bases, and 0.25% of carboxylic acids. The phenols distilled over the range 180–205° and contained phenol, all the cresols, xylenols, and pyrocatechol. The neutral oil gave liquid distillate fractions varying from d 0.79 to d 0.90, and higher-boiling fractions containing large quantities of paraffin wax and “resins.”

C. IRWIN.

Report of test by the Director of Fuel Research on the Crozier retort installed by Mineral Oils Extraction, Ltd., at Wembley. ANON. (Dept. Sci. Ind. Res., Fuel Res. Bd., 1927, 21 pp.).—The retort is of cast iron, and consists of two parallel walls 1 ft. 3 in. apart joined by semi-circular ends; the height is 15 ft. and the greatest width 5 ft. At each of five levels are three inclined cross-flues passing from one flat side of the retort to the other. Offtakes, which are carried to the centre of the retort and are hooded, are situated at four different levels. The material to be carbonised is fed into two hoppers at the top of the retort, and after passing through is extracted into a coke chamber by means of a rotating helix. The retort is enclosed in a brickwork chamber divided into compartments through which pass heating gases supplied from a furnace burning either solid or gaseous fuel. After condensation of the tar and liquor the gas is oil-scrubbed to recover the light spirit therein, and the greater part of the scrubbed gas is returned to the furnace for heating the retort. The coal used for the test was a Scottish splint coal (Tullygarth), and the throughput was about 4 tons per day. Some difficulty was encountered owing to holding up of the charge in the retort. The temperature of carbonisation varied from about 800° at the base of the retort to about 280° at the top. The yields of products per ton of dry coal were: coke, 15.4 cwt.; gas, 7390 cub. ft. of 323 B.Th.U./cub. ft.; tar, 16.4 gals.; spirit (crude), 0.8 gal.; ammonium sulphate, 9.1 lb. The coke was not completely carbonised, the volatile matter being 14.6%. It gave a satisfactory, but not quite smokeless, fire in a domestic grate.

A. B. MANNING.

Phenols in petroleum distillates. LER. G. STORY and R. D. SNOW (Ind. Eng. Chem., 1928, 20, 359—364).—The disposal of phenols which gradually accumulate in the alkaline solutions used for petroleum washing presents some difficulty, especially with cracked distillates, where the quantity is larger. A sample of such a caustic soda solution was acidified and distilled, and the distillate again extracted with caustic soda, acidified, and the phenols obtained were refractionated. The b.p. of the product ranged from 192° to 299°. It contained small proportions of cresols. Tests for other specific compounds gave negative results. A phenol mixture of lower b.p. range was extracted from low-boiling cracked distillate. With straight-run distillates the yields of phenols were very low, but the cresols were again identified. The general character of these phenols is similar to that of the phenols found in low-temperature tar. The quantity even in cracked distillates is usually less than 0.01%. C. IRWIN.

Carbon dioxide recorder. GORDON and LEHMANN. **Aluminium vessels.** BUSCHLINGER.—See I. **Blast-furnace gas.** HARBORD.—See X. **Graphite for electrotpe.** WINKLER and BLUM.—See XI.

PATENTS.

Coking retort oven. H. KOPPERS, Assr. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,664,632, 3.4.28. Appl., 31.8.21. Ger., 7.11.19).—An inclined retort is provided with vertical heating flues, of which those arranged alongside the lower part of the retort have their lower ends lying in a line parallel to the retort, whilst those alongside the upper part have their lower ends horizontal. Each set of flues communicates with a corresponding regenerator below, that for the lower flues having its upper part arranged stepwise, each step being directly connected with a vertical flue. Above the flues and connected with them runs a waste-gas collecting channel, over the horizontal portion of which a charging platform is constructed. A. B. MANNING.

Coking retort oven. [A] N. G. PARRY, [B] J. VAN ACKEREN, Assrs. to KOPPERS Co. (U.S.P. 1,664,633 and 1,664,649, 3.4.28. Appl., [A, B], 6.9.21. [B] Renewed 29.1.26).—(A) A coke oven consists of a series of coking chambers with heating walls constituted of vertical combustion flues disposed in pairs. Vertical outflow channels in each heating wall are connected with a pair of combustion flues, the individual outflow channels of one heating wall being connected, respectively, with those of an adjacent heating wall by ducts beneath the chambers. Crosswise regenerators parallel with the coking chambers are divided into inflow and outflow operating pairs, each regenerator of each pair being connected with the combustion flues of a pair of adjacent heating walls. (B) The coking chambers are heated by vertical combustion flues, with each pair of which vertical outflow channels are connected. The outflow channels of alternate heating walls are connected with the corresponding outflow channels of intermediate heating walls by ducts beneath the chambers. Crosswise regenerators are disposed as in (A), both regenerators of each pair corresponding to and being connected with combustion flues of only a single heating wall.

A. B. MANNING.

Manufacture of fuel briquettes. A. L. STILLMAN, Assr. to GEN. FUEL BRIQUETTE CORP. (U.S.P. 1,664,998, 3.4.28. Appl., 21.1.27).—A mixture of bituminous coal and a bituminous binder is subjected to an intense grinding operation at a temperature above the fusion point of the binder and in the presence of water vapour, until volatile constituents are liberated from the coal and dissolve in the molten binder. A. B. MANNING.

Production of hard low-temperature coke. K. JAHNKE (U.S.P. 1,665,665, 10.4.28. Appl., 19.10.26. Ger., 2.5.24).—A mixture of pulverised coal, spathic iron ore, and a bituminous material is subjected to a low-temperature carbonisation process. A. R. POWELL.

Manufacture of active carbon from soot. G. HUGHES. From T. FUJISAWA (B.P. 287,982, 13.1.27).—Soot obtained by the incomplete combustion of oils, fats, hydrocarbon gases, etc. is heated in a thin layer to a dull red heat and stirred in contact with air until all the volatile impurities have been removed. It is then quickly transferred to a closed vessel and cooled.

A. B. MANNING.

Cooling of coke or like material and utilising the sensible heat thereof. FRANKFURTER GAS-GES., F. P. TILLMETZ, and E. SCHUMACHER (B.P. 280,514, 14.10.27. Ger., 11.11.26).—The sensible heat of incandescent coke is utilised in stages by an arrangement in which the coke passes over successive grates through which inert gases are blown. *E.g.*, in the higher stage, during which the coke cools from about 1000° to 700°, the hot gases may be conducted through a high-pressure boiler, and in the lower stage, from about 700° to 300°, the gases may traverse a low-pressure boiler or water preheater. The first grate may be a stepped grate and the second either a stepped or a travelling grate. The coke leaving the last grate passes into a bunker in which it is completely cooled by warm water or low-pressure steam.

A. B. MANNING.

Preparation of charges for combustion in internal-combustion engines. G. B. ELLIS. From MOORE INVENTIONS CORP. (B.P. 287,996, 7.2.27).—In order to secure complete combustion of the fuel in an internal-combustion engine the charge is formed by producing a flow of mixed fuel and air and delivering into admixture therewith a flow of exhaust gas and air, the temperature of the charge being controlled by the temperature of the air mixed with the exhaust gas. Hot air may be mixed with exhaust gases at low engine compression pressures and relatively cold air at higher compression pressures. The charge is compressed and ignited in a cylinder having inclined vanes adjacent to its head, the gases as they pass these vanes being thereby given a turbulent motion.

A. B. MANNING.

Balanced motor fuel. E. F. CHANDLER (U.S.P. 1,665,621, 10.4.28. Appl., 23.12.24).—A motor fuel comprises a liquid hydrocarbon, an oxide of nitrogen, and a solvent for the latter.

A. R. POWELL.

Distillation of coal and similar carbonaceous substances. LOW TEMPERATURE CARBONISATION, LTD., and C. H. PARKER (B.P. 287,584—7 and 287,830, 25.9.26).—(A) A retort for the production of smokeless fuel by the distillation of coal at low temperatures consists of

a number of vertical chambers, substantially rectangular in cross-section and slightly tapered (*e.g.*, $4\frac{1}{2} \times 11$ in. at the upper end, $5\frac{1}{2} \times 12$ in. at the lower end, and 9 ft. high), formed in a single casting and arranged to present a large external heating surface. The upper and lower ends of the casting have flanges so that a hood may be fitted above the retorts and a hinged bottom plate below. (b) In a process for the distillation of coal in two stages, in the second of which the material is transferred to a retort of considerable volume, water is passed into this retort and vaporises therein, the material being thus cooled and quenched in an atmosphere of steam at a slight pressure. (c) Coal is carbonised at low temperatures by being filled into cylindrical containers and then feeding these into a retort through which they pass by rolling on inclined tracks from the charging to the discharging positions. The containers are open at both ends and are provided with perforated covers. The retorts are made up of a number of connected sections of opposite inclination one above the other, which the filled containers traverse in succession. Each section of retort is mounted in a corresponding retort chamber; heating gases circulate through the chambers, which communicate with one another. The volatile products of distillation are withdrawn from a point near the uppermost end of the retort, or from points near the junctions of the sections. Automatically operating means are provided for filling the containers, charging them into and discharging them from the retort, sealing the retort, and discharging the coke from the containers. (d) In the distillation of coal described under (c) the material is subjected to a preliminary drying by causing the filled containers to traverse a chamber through which hot waste gases pass. (e) In an apparatus for the distillation of coal in which the process is carried out in two stages, first in an externally heated upper retort and then in a separate lower retort without further application of heat, means are provided for the withdrawal of gas from the upper end of the lower retort and conducting it through an auxiliary ascension pipe up and into the main ascension pipe at a point behind the anti-dip valve. A. B. MANNING.

Distillation and gasification of solid carbonaceous material. A. K. CROAD. From ZIELEY PROCESSES CORP. (B.P. 287,999, 14.2.27).—Liquid hydrocarbons of low mol. wt., *e.g.*, gasoline, naphtha, are produced from coal or other carbonaceous material by impregnating the latter with a chemical such as sodium chloride, and subjecting it to distillation and gasification in a current of air and steam at 1370° . The sodium chloride, which is resolved into its constituents at this temperature, acts only as a catalyst. A. B. MANNING.

Distillation of solid carbonaceous or bituminous substances. W. E. EVANS. From KOHLENVEREDLUNG A.-G. (B.P. 287,627, 22.12.26).—The carbonaceous material in the form of a fine powder is carried in a continuous stream with the heating gas, the initial temperature of which is above about 800° , and is maintained in contact therewith for only the short time necessary for the low-temperature distillation of the fuel. Although the initial temperature of the heating gas is above 800° the heat taken up by the preliminary drying of the fuel

particles causes this to fall considerably before actual carbonisation commences, so that the latter process then occurs without overheating of the fuel or cracking of the tar vapours. A. B. MANNING.

Distillation of solid carbonaceous materials. H. NIELSEN and B. LAING (B.P. 287,381, 4.12.26, 21.2. and 18.5.27).—In the distillation of bituminous coal etc. cracking of the oil vapours evolved is prevented by so regulating the heating that the temperature difference between the periphery and the centre of any coal particle or any suitable thickness of fuel bed never exceeds 150° . The material may be crushed below 2-in. mesh and passed continuously, during about 2 hrs., through zones of progressively higher temperature produced by heating gases having a temperature gradient of approximately 650° down to 110° , the oil vapours being withdrawn from the apparatus at approximately the temperatures at which they are evolved. The process may be carried out by the use of a large volume of an inert gaseous heating medium as in B.P. 287,037 (B, 1928, 356) and yields products similar to those therein described. A. B. MANNING.

Apparatus for the low-temperature distillation or coking of fuels. J. PLASSMANN (B.P. 287,313, 9.3.27).—A retort comprises a number of superposed annular distillation chambers alternating with heating chambers. The former are closed but the latter are open and in communication with one another at the outer periphery. A centrally disposed shell, which can be rotated, supports the charging and discharging devices, and also the internal peripheral closing means for the distillation chambers. The material is charged into the distillation chambers from a central feeding shaft by means of radially disposed screw conveyors. The heating gases are circulated through the heating chambers by fans. The above system of distillation may be combined with that described in B.P. 240,800 (B., 1926, 228), to form a single unit. A. B. MANNING.

Gas producer. C. W. LUMMIS, Assr. to MORGAN CONSTRUCTION Co. (U.S.P. 1,666,604, 17.4.28. Appl., 19.12.21).—The shell of a gas producer and the water-seal pan are connected and arranged so that they can be rotated together. An ash-plough is supported on the water-seal pan, and, by means of a pivot through the centre of the seal, can be rotated at a different speed from the pan and shell. T. A. SMITH.

Apparatus for making carburetted water-gas. HUMPHREYS & GLASGOW, LTD., Assecs. of C. S. CHRISMAN (B.P. 282,034, 21.11.27. U.S., 10.12.26).—In a carburetted water-gas set a regenerator is interposed between the superheater and the wash box in order to recover the sensible heat of the carburetted water-gas and to utilise it for preheating the air or superheating the steam supplied to the generator. The regenerator may be provided with a shaking grate carrying a bed of clinker, or may be filled with chequer brick. A. B. MANNING.

Manufacture of mixed water-gas and distillation gas from fuels of volatile content. HUMPHREYS & GLASGOW, LTD., Assecs. of C. S. CHRISMAN (B.P. 279,060, 27.9.27. U.S., 13.10.26).—A plant for the complete gasification of fuels of high volatile content consists of

two generators, two stoves for heat storage, a tubular heater, and a wash box. The generators are divided into a lower gasification zone and an upper carbonisation zone. The first operation of the cycle consists of a simultaneous blasting in the gasification zones of both generators, the resulting blow gases passing through the stoves, in which most of their heat becomes stored. The second operation consists of up-steaming both zones in one generator and simultaneously down-steaming the lower zone in the other, and passing the water-gas produced in the latter through one of the heated stoves and then through the upper zone of the former generator together with the water-gas generated in the lower zone thereof. The resulting mixed water-gas and distillation gas is collected. The blow gases entering the stoves may be burnt therein with secondary air preheated in the tubular heater by waste gases from the stoves. Some operating cycles slightly modified from the above are described.

A. B. MANNING.

Production of gas. R. LEDERER (B.P. 270,318, 28.4.27. Austr., 28.4.26.).—A mixture of water-gas and coal gas is produced from a bituminous fuel, and coke obtained as a by-product, by the use of a generator and a separate coking chamber so arranged that the producer gas formed during the "blow" is led either directly, or after partial combustion, first through the lower zone of the coking chamber containing fuel which has been distilled, for the purpose of hardening the coke therein, and then through one or more heat accumulators. The mixture of water-gas and coal gas produced during the "run" is conducted through the coking chamber, or through a heat accumulator, in order to decompose the tar vapours in the coal gas and thereby increase the calorific value of the gas produced.

A. B. MANNING.

Manufacture of gas. J. HANSFORD and J. S. HUGHES (B.P. 287,213, 13.12.26.).—A mixture of liquid or powdered fuel is injected into a combustion chamber wherein the fuel undergoes partial combustion in the presence of iron or other metal shavings. The gas may be enriched by feeding oil into the combustion chamber, or its hydrogen content may be increased by blowing in steam. The burner comprises two semicircular nozzles arranged to produce a conical combustion zone, and may be placed in the lower or the upper end of the combustion chamber. A valve operated by the pressure in the combustion chamber controls the supply of fuel to the burner.

A. B. MANNING.

Drying of fuel gases. D. M. HENSHAW, C. COOPER, and W. C. HOLMES & Co., LTD. (B.P. 287,678, 5.2.27.).—The gases are partially dried by treatment with a hygroscopic liquid at the ordinary temperature, and then subjected to a similar drying process with the application of artificial cooling. The liquid is reconcentrated either periodically or continuously. Cooling may be effected by having two vessels in communication with one another, one containing a hygroscopic liquid and the other water, and maintaining a reduced pressure in the apparatus, evaporation of the water producing the required reduction of temperature. Means may be provided for agitating the water in the apparatus and so promoting evaporation.

A. B. MANNING.

Recovery of phenol from waste water containing ammonia. F. RASCHIG (B.P. 287,226, 15.12.26.).—The removal of phenol from waste water by passage of the latter through a column counter-current to a suitable extracting liquid, *e.g.*, benzol, is made very efficient by the use of 15-mm. Raschig rings as filling material for the column. The waste water is preferably heated to 50–60° by using it for condensing the benzol vapour in the subsequent distillation of the benzol-phenol mixture.

A. B. MANNING.

Production from coal tar of neutral lubricating oil free from salts, stable in the air, and capable of admixture with mineral oils. "REN" MINERALÖL-GES. STEPHAN, BOOK, & ZIEGLER (G.P. 445,679, 10.4.24. Addn. to G.P. 444,958; B., 1928, 116).—The vapours generated during the distillation of coal tar pass over solid alkali hydroxides, and, after the distillate has been cooled rapidly to a low temperature, preferably *in vacuo*, any constituents which settle out are removed.

L. A. COLES.

Conversion of petroleum hydrocarbons. W. M. CROSS (U.S.P. 1,666,119, 17.4.28. Appl., 20.7.25).—Oil is heated in a tubular still, vaporised in a separate chamber, and the unvaporised residue is returned to the still for re-treatment; the re-treated oil is passed to a separate vaporising and condensing apparatus. The degree of heating to which the charging stock and re-cycled oil are subjected is controlled by adjusting their rates of circulation through the still.

C. O. HARVEY.

Separation of gasoline from crude [mineral] oil. Distillation of mineral oil. A. E. PEW, JUN., and H. THOMAS, ASSRS. to SUN OIL CO. (U.S.P. 1,666,300—2, 17.4.28. Appl., 23.2.24).—(A) The heated oil is sprayed into a chamber at successively higher elevations and diminishing temperatures, and gives up its heat to a downflowing stream of oil with which it is not in actual contact. (B) The oil is heated to a temperature adequate for the degree of vaporisation required, and is sprayed in a number of streams into a vaporising chamber. The streams are recombined and the operation is repeated a number of times at progressively increasing heights in the chamber, and correspondingly decreasing temperatures. The unvaporised oil is finally allowed to flow down the column under conditions favouring heat-interchange, but out of direct contact with the hot oil streams. (C) The downflowing stream of oil is augmented by an independent stream of mineral oil which finally passes to the heating zone.

C. O. HARVEY.

Distillation of oils. A. E. HARNSEBERGER, ASSR. to PURE OIL CO. (U.S.P. 1,666,597, 17.4.28. Appl., 12.11.26).—Mineral oil is submitted to vacuum distillation in a pipe still, which is built of progressively increasing cross-sectional pipe area from the inlet to the outlet ends, to allow for the increasing volume of the oil and vapours and to reduce back-pressure.

T. A. SMITH.

Distillation of [mineral] oils. C. B. BUERGER, ASSR. to GULF REFINING CO. (U.S.P. 1,666,042, 10.4.28. Appl., 28.8.23).—The oil in a horizontal cylindrical still, fitted with a system of partitions and a steam jet, is

maintained in a state of constant motion, deposition of carbon being thereby prevented. C. O. HARVEY.

Distillation of hydrocarbon oils. W. P. DEPPÉ and L. L. SUMMERS (U.S.P. 1,666,051, 10.4.28. Appl., 5.1.24).—Crude oils and mixtures of oils with other liquids are passed through tubular stills at progressively increasing temperatures, and the mixture of vapours and liquid is subjected to series of dephlegmating operations. The resulting vapours of predetermined composition are combined, and the residues pass back for re-treatment. C. O. HARVEY.

Refining of heavy mineral oils by means of sulphur dioxide in a continuous operation. L. EDELEANU, K. PFEIFFER, K. GRESS, and P. JODECK (U.S.P. 1,666,560, 17.4.28. Appl., 25.6.27. Ger., 12.6.24).—Mineral oil and liquid sulphur dioxide are continuously run into a mixing vessel fitted with stirring device. The mixture is passed to a larger vessel, being delivered about the middle of the latter vessel, which is full of liquid. Oil is continuously drawn from the top of the separating vessel and sulphur dioxide solution from the bottom. T. A. SMITH.

Manufacture of a gasoline substitute. J. B. NEUENDORFF (U.S.P. 1,666,976, 24.4.28. Appl., 6.8.26).—Petroleum distillates for use in internal-combustion engines are subjected, in a liquid state, to the action of clear lime water. F. G. CROSSE.

Recovery of aluminium chloride from sludge. P. DANCKWARDT (U.S.P. 1,665,406, 10.4.28. Appl., 23.4.27).—Aluminium chloride "sludge" and a gas are injected into a mass of molten metal, and the aluminium chloride is condensed. C. O. HARVEY.

Anti-sludging means for oil. D. C. COX, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,665,845, 10.4.28. Appl., 28.9.26).—Mineral oil is heated in contact with fuller's earth in an electrically heated vessel. T. A. SMITH.

Manufacture of carbonised fuel. W. W. STRAF-FORD, Assr. to FUELITE Co., LTD. (U.S.P. 1,667,906, 1.5.28. Appl., 21.4.24. U.K., 15.5.23).—See B.P. 221,526; B., 1924, 932.

Manufacture of valuable products from combustible materials. A. H. PEHRSON, Assr. to DUNFORD & ELLIOTT (SHEFFIELD), LTD. (U.S.P. 1,667,217, 24.4.28. Appl., 13.8.23. Swed., 19.8.22).—See B.P. 202,625; B., 1924, 859.

Gas-treating process. C. LOURENS, Assr. to GEN. NORIT Co., LTD. (U.S.P. 1,667,426, 24.4.28. Appl., 11.11.22. Holl., 14.11.21).—See B.P. 188,666; B., 1923, 914 A.

Exothermic gas reactions (B.P. 275,983).—See I. **Rock asphalt** (U.S.P. 1,664,846). **Bituminous emulsion** (Swiss P. 119,219). **Bituminous product** (U.S.P. 1,665,881). **Pavements** (B.P. 287,799).—See IX. **Furnace for shales** (U.S.P. 1,665,546). **Recovery of reaction products** (B.P. 263,859 and 288,056).—See XI. **Lamp blacks etc.** (B.P. 270,658).—See XIV.

III.—ORGANIC INTERMEDIATES.

Synthesis of alcohols higher than methyl alcohol from carbon monoxide and hydrogen. K. FROLICH and W. K. LEWIS (Ind. Eng. Chem., 1928, 20, 354—359).—The comparative efficiency of various catalysts in the formation of higher alcohols from carbon monoxide and hydrogen was investigated, the pressure used being 204 atm. Steel or chromium-steel turnings impregnated with potassium hydroxide gave unsatisfactory results, the loss as gaseous products in most cases varying from 30 to 70%. A low-chromium steel with a minimum of added alkali appeared the most effective. The highest oil yield obtained was 13.4%, and increase of oil yield was associated with increase of gas losses. In combination with a methyl alcohol catalyst, gas losses were less, but only traces of oil were obtained, the product being a mixture of methyl alcohol and higher alcohols. Metallic oxide catalysts, such as a mixture of zinc oxide, chromic oxide, and barium hydroxide on copper, which yield methyl alcohol at 300—350°, produced higher alcohols at 450—500°. The best yield recorded, however, was 13.9%, chiefly propyl alcohol. The loss was rather less than with iron-alkali catalysts, and the product was largely alcoholic in place of the complex mixture of alcohols, aldehydes, ketones, etc. obtained in the other case. C. IRWIN.

Phenols in petroleum distillates. STORY and SNOW.—See II. **Furfuraldehyde from wood.** RITTER and FLECK.—See V. **Tests for ether.** MIDDLETON and HYMAS.—See XX. **Carbon disulphide.** EGGERT.—See XXIII.

PATENTS.

Oxidation of alcohol. S. GOLDSCHMIDT (U.S.P. 1,666,447, 17.4.28. Appl., 16.12.27. Ger., 12.11.26).—Silver at 380—440° catalytically converts a mixture of ethyl alcohol and air into acetaldehyde and acetic acid. B. FULLMAN.

Manufacture of acetaldehyde from gaseous mixtures containing acetylene. I. G. FARBENIND. A.-G. (B.P. 278,324, 2.8.27. Ger., 28.9.26).—Acetylene, in gas mixtures containing it, is converted into acetaldehyde by treating the mixture, in a finely-divided state (e.g., by passage through microporous glass plates), under at least 2 atm. pressure, and (where the acetylene content is low) at 80° or above, with acid solutions containing mercury salts. The aldehyde is separated from the gas (e.g., by passage through water) prior to expansion of the latter. B. FULLMAN.

Manufacture of formic acid. G. H. BUCHANAN and J. L. OSBORNE, Assrs. to AMER. CYANAMID Co. (U.S.P. 1,666,437, 17.4.28. Appl., 10.12.23).—Formic acid is added to an alkaline-earth formate, and the mixture treated with a non-volatile acid capable of decomposing it; the formic acid formed is then boiled off. H. ROYAL-DAWSON.

Extraction of butyric acid. Soc. DES BREVETS ÉTRANGERS LEFRANC ET CIE. (B.P. 276,617, 11.2.27. Fr., 27.8.26).—Alkali or alkaline-earth butyrates are treated with a slight defect of hydrochloric acid. The liquid separates into two layers. The upper contains

the greater part of the butyric acid in 85—90% concentration, which may be further concentrated as usual. The lower contains the metallic chloride and some butyric acid, which may be recovered, *e.g.*, by distillation with superheated steam.

B. FULLMAN.

Coagulation or peptisation of products containing unsaturated carbon compounds. L. AUER (B.P. 287,943, 30.9.26).—Isocolloid substances containing unsaturated carbon compounds undergo a physical or physico-chemical change when heated with "electrolytes," under which term are included salts, metal derivatives of organic substances, and solid organic acids of the aromatic and lower aliphatic series; liquid inorganic or organic acids may be used in conjunction with these. The "electrolyte" is added dry to the isocolloid and is dissolved in it with the aid of heat and, if desired, by grinding. The treatment may be followed or accompanied by a treatment with a gas or gases at reduced, ordinary, or increased pressure. The result is to convert liquid starting materials (vegetable oils, liquid unsaturated hydrocarbons, etc.) into pasty or hard solids, and solid starting materials (resins etc.) into pasty or thin liquids; rubber, tars, asphalts, etc. may also be used. The properties of the final product may be modified by adding to the starting materials purely organic substances (phenols, naphthols, naphthalene, chloroform, acetone, alcohols); siccatives may also be added. The products may be vulcanised during or after the modifying process, *e.g.*, by means of sulphur chloride, sulphur, or sulphur and an accelerator. The examples refer to linseed oil and rosin, the products being useful as rubber substitutes, linoleums, varnishes, etc. The process appears to be physical rather than chemical, and is reversed if by prolonged washing or grinding with a solvent the "electrolyte" is moved. C. HOLLINS.

Manufacture of alkyl β -halogenoethyl ketones. SCHERING-KAHLBAUM A.-G. (B.P. 282,412, 14.12.27. Ger., 15.12.26).—Ethylene reacts with acyl halides in presence of aluminium chloride or bromide to give alkyl β -halogenoethyl ketones. Examples are *methyl β -chloroethyl ketone*, b.p. 50—55°/16 mm.; *methyl β -bromoethyl ketone*, b.p. 55—60°/15 mm. C. HOLLINS.

Manufacture of 1-amino-8-naphthoic acid. R. HERZ and F. SCHULTE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,668,148, 1.5.28. Appl., 9.5.27. Ger., 29.11.24).—See B.P. 276,126; B., 1927, 808.

Continuous dehydration of volatile fatty acids. E. RICARD, Assr. to SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (U.S.P. 1,668,380, 1.5.28. Appl., 26.12.24. Belg., 28.12.23).—See B.P. 226,822; B., 1925, 737.

Manufacture of chlorinated amines from chlorinated hydroaromatic ketimino-compounds [chloro-ketimides]. T. VOLTZ, Assr. to DURAND & HUGUENIN SOC. ANON. (U.S.P. 1,667,336, 24.4.28. Appl., 21.5.23. Ger., 3.6.22).—See B.P. 217,753; B., 1924, 769.

Condensation products of anthraquinone (B.P. 263,178).—See IV.

IV.—DYESTUFFS.

Some azo dyes soluble in non-aqueous solvents. C. E. MAY and H. HUNT (Ind. Eng. Chem., 1928, 20, 384—388).—A number of new azo dyes, soluble in hydro-

carbons, linseed oil, and carbon tetrachloride, but insoluble in water, have been prepared. In the following list the name of the diazotised intermediate is followed by that of the substance with which it was coupled, and the m.p. of the resulting dyestuff: 3-*o*-xylydine, β -naphthol, 125—130°; 3-*o*-xylydine, 1-amino- β -naphthol, 140—145°; 4-*m*-xylydine, β -naphthol, 132—136°; 4-*m*-xylydine, 1-amino- β -naphthol, 140—144°; 4-*m*-xylydine, phenol, 172—174°; 4-*m*-xylydine, resorcinol, 275—280°; 4-*m*-xylydine, guaiacol, 110—112°; *p*-xylydine, β -naphthol, 115—120°; *p*-xylydine, 1-amino- β -naphthol, 105—106°; aminoazo-4-*m*-xylene, β -naphthol, 145—150°; aminoazo-4-*m*-xylene, 1-amino- β -naphthol, 140—150°; aniline, α -naphthol, 148—150°; aniline, 1-amino- β -naphthol, 110—115°; aniline, 1-nitroso- β -naphthol, 106°; *o*-aminoazotoluene, salicylic acid, 125—130°; *o*-aminoazotoluene, resorcinol, above 360°; *o*-aminoazotoluene, α -naphthol, 153—156°; *o*-aminoazotoluene, phenol, 290—295°; aminoazo-*p*-xylene, β -naphthol, 180—185°; aminoazo-*p*-xylene, 1-amino- β -naphthol, 110—115°; β -naphthylamine, α -naphthol, 146°; β -naphthylamine, dimethylaniline, 153—158°; benzidine, 2 mols. of α -naphthol, 255—260°; benzidine, 1 mol. of phenol and 1 mol. of α -naphthol, above 360°; benzidine, 2 mols. of α -naphthylamine, above 360°; *o*-anisidine, 1-amino- β -naphthol, 135—140°; aminoazo- β -naphthylamine, β -naphthol, 107—109°; commercial xylydine and α -naphthol, 113—118°; 6-nitro-*as-m*-xylydine, β -naphthol, 180—185°; 1-amino- β -naphthol, dimethylaniline, 135—140°. The dyestuffs had the expected composition, except for the last-mentioned, which had apparently acquired a nitroso-group. The relative solubilities of these and other similar known dyes in toluene and in carbon tetrachloride, expressed as the smallest amount of solvent which dissolved completely 0.5 g. of the sample, were determined. Reduction of these dyes was found to be impossible with stannous chloride and hydrochloric acid, but was readily accomplished by means of sodium amalgam and alcohol at 100°. On reduction of the dye from 4-*m*-xylydine and 1-amino- β -naphthol by this method, the diamidonaphthol first produced was very readily oxidised and the substance isolated was a dark red powder, apparently 4-amino-1:2-naphthaquinone (m.p. 136°). When this substance was diazotised and the diazonium salt coupled with β -naphthol a bright red dye resulted.

W. J. POWELL.

PATENTS.

Manufacture of new dyes of the anthanthrone series. L. CASSELLA & Co., G.M.B.H. (B.P. 287,020, 3.11.26. Addn. to B.P. 260,998; B., 1928, 225).—The bromination and/or chlorination of anthanthrone is performed in presence of excess of liquid bromine or sulphuryl chloride as diluent; alternatively, anthanthrone is treated with gaseous halogen, either reaction being assisted, if desired, by addition of a carrier (iodine, ferric chloride). Orange vat dyes similar to those of the prior patent are obtained. C. HOLLINS.

Manufacture of condensation products of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 263,178, 17.12.26. Ger., 17.12.25).—*o*-Aminothiolarthraquinones are condensed with halogenohydrins (ethylene chlorohydrin, propylene chlorohydrin, α -chloro-

hydrin) to give thioethers (not thiomorpholines), which are acetate silk dyes and also intermediates. Examples are the β -hydroxyethyl thioethers of 1-amino-2-thiolanthraquinone (m.p. 171—173°), 2-chloro-1:4-diamino-3-thiolanthraquinone, 1-amino-4-*p*-toluidino-2-thiolanthraquinone, etc.

C. HOLLINS.

Manufacture of condensation products and vat dyes of the benzanthrone series. I. G. FARBENIND. A.-G., Assces. of FARBW. VORM. MEISTER, LUCIUS, & BRÜNING (B.P. 261,757, 17.11.26. Ger., 17.11.25. Addn. to B.P. 249,891; B., 1927, 647).—Halogenated benzanthranyl mercaptans, sulphides, or disulphides are condensed with phenols or mercaptans, and the products give by alkaline fusion reddish-blue to violet vat dyes. Examples are dibromo-1:1'-dibenzanthranyl sulphide with phenol, β -naphthol, or *p*-thiocresol.

C. HOLLINS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G. (B.P. 273,656, 7.3.27. Ger., 1.7.26).—Vat dyes are obtained by treating with acid condensing agents (boric and sulphuric acids, aluminium chloride) the benzanthranyl aminoanthraquinones obtained from α -aminoanthraquinones and 4-chloro- or 4:8-dichloro-benzanthrone. 4'-Benzanthranyl- α -aminoanthraquinone gives a bright orange-red vat dye, 4:8-di- α -anthraquinonylaminobenzanthrone a brown, 6-chloro-4'-benzanthranyl-1-aminoanthraquinone a red-brown, 6-amino-4'-benzanthranyl-1-aminoanthraquinone a violet-brown, 8'-chloro-4'-benzanthranyl-1-amino-4-methoxyanthraquinone a brown.

C. HOLLINS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G. (B.P. 278,651, 17.3.27. Ger., 8.10.26).—Aminobenzanthrone is diazotised in concentrated sulphuric acid, and the mixture diluted and boiled; the hydroxybenzanthrone, m.p. 287—288°, so produced is alkylated (e.g., with methyl toluenesulphonate to form methoxybenzanthrone, m.p. 167—168°), and finally fused with alkali. In the case of methoxybenzanthrone the green vat dye obtained is identical with that of B.P. 181,304 and 218,255.

C. HOLLINS.

Manufacture of vat dyes of the dibenzanthrone series. I. G. FARBENIND. A.-G. (B.P. 262,819, 13.12.26. Ger., 14.12.25).—3:3'-Dibenzanthronyls, unsubstituted in the 2:2'-positions, are reduced in alkaline media to give leuco-vat dyes. For example, 3:3'-dibenzanthronyl is boiled with dilute sodium hydroxide, and sodium formaldehydesulphoxylate is gradually added; the resulting vat is oxidised with hypochlorite to precipitate the new vat dye.

C. HOLLINS.

Manufacture of vat dyes of the dibenzanthrone series. I. G. FARBENIND. A.-G. (B.P. 263,861, 30.12.26. Ger., 30.12.25. Addn. to B.P. 218,255; B., 1925, 538).—The dyes of the prior patent are obtained in better yield and purer condition by alkaline fusion of suitable dialkoxy-3:3'-dibenzanthronyls, e.g., 2:2'-dimethoxy-3:3'-dibenzanthronyl, m.p. 387—390°, obtained by heating with copper the 3-iodo-2-methoxybenzanthrone, m.p. 248°, prepared from 2-methoxybenzanthrone by nitration, reduction, and the Sandmeyer reaction.

C. HOLLINS.

Manufacture of grey to black vat dyes. I. G. FARBENIND. A.-G. (B.P. 264,502, 12.1.27. Ger., 12.1.26).—Aminodibenzanthrone is treated with methyl

sulphate or methyl alcohol and sulphuric acid at 170° in the absence of acid-binding agents to give a complex condensation product which dyes cotton grey to black shades from a blue vat.

C. HOLLINS.

Manufacture of azo dyes. J. R. GEIGY A.-G. (B.P. 275,223, 22.7.27. Ger., 29.7.26).—An amino-sulphobenzoic acid in which the sulphonic group is *ortho* to the amino-group is diazotised and coupled with the usual components (excluding H-acid and cyanuric derivatives). The dyes, especially those from 2-chloro-5-amino-4-sulphobenzoic acid, show superior fastness to light compared with the non-carboxylated dyes. The β -naphthol coupling gives a barium or calcium lake superior to Lake Red C; the γ -acid coupling is a bluish-red wool dye, little changed in shade by after-chroming.

C. HOLLINS.

Manufacture of trisazo dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 287,232, 16.12.26. Addn. to B.P. 248,230. Cf. F.P. 598,894; B., 1926, 311).—1:2-Aminonaphthyl ethyl ether is used as third component in place of the sulphonic acid of the prior patent, and/or other J-acid derivatives in place of J-acid and aryl-J-acids, the coupling of the end-component being effected in presence of pyridine. Examples are: aniline-2:5-disulphonic acid \rightarrow α -naphthylamine or its 7-sulphonic acid \rightarrow 1-amino-2-naphthyl ethyl ether \rightarrow phenyl-J-acid or ethyl-J-acid (greenish-blue); *o*-toluidine-3:5-disulphonic acid \rightarrow α -naphthylamine-7-sulphonic acid \rightarrow 1-amino-2-naphthyl ethyl ether \rightarrow 6-hydroxy-2-*m*-aminophenyl- α -naphthiminazole-8-sulphonic acid (from 1:2-diamino-5-naphthol-7-sulphonic acid; bluish-green) or 1-phenyl-3-methyl-5-pyrazolone (green); aniline-*o*-sulphonic acid \rightarrow α -naphthylamine-7-sulphonic acid \rightarrow 2-ethoxy- α -naphthylamine-7-sulphonic acid \rightarrow *m*-aminobenzoyl-J-acid (greenish-blue).

C. HOLLINS.

Manufacture of dyes containing chromium. I. G. FARBENIND. A.-G. (B.P. 262,418, 25.11.26. Ger., 1.12.25).—Chromable dyes are heated in open or closed vessels with chromic salts in presence of a solution of a mineral acid salt of a metal which does not combine with the dye (i.e., a non-mordanting metal). The shades obtained are different from those obtained in the absence of the non-mordant salt. E.g., the dye, 4-nitro-2-aminophenol-6-sulphonic acid \rightarrow β -naphthol, heated with chromium formate solution and sodium chloride, gives a black wool dye, whereas in the absence of sodium chloride the shade is violet brown. The amount of non-mordant salt must be at least equal to the weight of dye.

C. HOLLINS.

Manufacture of dyes [of the perylene series]. F. BENSA (B.P. 278,325, 9.8.27. Austr., 2.10.26).—When 3:9-dichloro-4:10'-diacetyl- (or dipropionyl-) perylene is heated with copper cyanide in boiling quinoline (or pyridine under pressure at 190—200°), the chlorine atoms are replaced by nitrile groups, and further condensation occurs with production of reddish-violet vat dyes. The starting materials are obtained from 3:9-dichloroperylene and acetyl and propionyl chlorides in presence of aluminium chloride at 15°.

C. HOLLINS.

Manufacture of triarylmethane dyes. BRIT. DYESTUFFS CORP., LTD., and T. A. SIMMONS (B.P. 287,995, 5.2.27).—Tetraethyldiaminobenzhydrol is isolated (from the oxidation product of the methane with lead peroxide) in the form of its 2:7-naphthalene-disulphonate, and this salt is condensed in sulphuric acid to the leuco-compound of Lissamine Green V, from which the dye is obtained in the usual manner.

C. HOLLINS.

Manufacture of colour lakes. I. G. FARBENIND. A.-G. (B.P. 270,750, 7.5.27. Ger., 10.5.26).—Colour lakes made from complex phosphorus, tungsten, molybdenum, and silicon acids and basic dyes are improved by heating in paste form under 2–4 atm. pressure either during or after the manufacture. The treatment may be carried out in presence of the usual substrates, dispersing agents, and, if desired, a further quantity of complex acid.

C. HOLLINS.

Manufacture of black copying colours. I. G. FARBENIND. A.-G. (B.P. 282,804, 28.12.27. Ger., 27.12.26).—A dialkylphenosafranine is diazotised and coupled with an alkylated or aralkylated 2-amino-*p*-cresol (cf. B.P. 283,777; B., 1928, 226), *e.g.*, 2-ethyl-amino- or 2-benzylamino-*p*-cresol.

C. HOLLINS.

Manufacture of vat dyes. J. G. DINWIDDIE, Assr. to E. I. DU PONT DE NEMOURS & Co. (Re-issue 16,939, 24.4.28, of U.S.P. 1,558,252, 20.10.25).—See B., 1925, 983.

Manufacture of azo dyes. H. FRITZSCHE, E. KRUMMENACHER, H. GUBLER, and O. KAISER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,667,312, 24.4.28. Appl., 21.12.23. Fr., 9.1.23).—See B.P. 209,723; B., 1925, 437.

Manufacture of azo dyes. A. L. LASKA and A. ZITSCHER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,667,667, 24.4.28. Appl., 10.9.25. Ger., 3.10.24).—See G.P. 430,580; B., 1927, 275.

Manufacture of [azo] dyes [containing metal]. F. STRAUB and H. SCHNEIDER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,667,333, 24.4.28. Appl., 12.3.26. Switz., 28.3.25).—See B.P. 249,884; B., 1927, 674.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Wetting of pine wood. C. G. SCHWALBE and W. LANGE (Papier-Fabr., 1928, 26, 238–241).—Pine heartwood absorbs a much larger quantity of magnesium bisulphite solution than sapwood in the same time, whereas with sodium bisulphite the amounts taken up are not markedly different. Sapwood in cylindrical form (3 cm. long, 3 cm. diam.) was shaken for 8 hrs. at 35–70°, and allowed to remain for 24 hrs. with solutions of sodium sulphite, sodium bisulphite, and sodium bisulphite containing free sulphurous acid; the quantities of solution absorbed per 100 g. of air-dry wood were 104.2, 85.0, 120.2 g., indicating that the presence of free sulphurous acid is necessary to ensure rapid wetting of the chips. The amount of water taken up under the same conditions was only 89.0 g., that of sulphurous acid solution (4.5% SO₂) 128.7 g., and sodium hydroxide solution 134.0 g. On cutting up the chips which had

been immersed in sodium sulphite solution, exterior portions were found to contain a solution of the same composition as the bulk, whilst inner portions were acid to litmus and contained a solution with a considerable sodium bisulphite content. Inner portions of chips after immersion in sodium bisulphite solution with or without excess of sulphurous acid, contained sulphurous acid solution only. This selective absorption did not take place when corresponding calcium and magnesium salts were used in place of sodium salts. W. J. POWELL.

Furfuraldehyde and carbon dioxide from wood before and after chlorination. G. J. RITTER and L. C. FLECK (Ind. Eng. Chem., 1928, 20, 371–373).—A study of the Cross and Bevan method of cellulose isolation (alternate treatment of wood with chlorine and sodium sulphite solution). No substances capable of producing furfuraldehyde on boiling with dilute hydrochloric acid were formed during the chlorination of a sample of wood, but a considerable amount of a substance which yielded carbon dioxide with boiling dilute hydrochloric acid was obtained, chiefly during the first chlorination, this material differing from oxycellulose in that it was soluble in sulphite solution and yielded no furfuraldehyde. These results indicate that there is no danger of forming oxycellulose in the determination of cellulose by the Cross and Bevan method. The original wood yielded 0.86% of carbon dioxide with boiling dilute hydrochloric acid, and 56.6% of the unstable pentosans present were rendered soluble in sulphite solution during the first chlorination.

W. J. POWELL.

Micro-method for the determination of the nitrogen content of nitrocellulose. Fractionation of nitrocellulose by diffusion. D. KRÜGER (Z. angew. Chem., 1928, 41, 407–408).—A sample of nitrocellulose (5–10 mg.) is moistened with a small quantity of alcohol and hydrolysed by heating at 50–60° with 5 c.c. of 30% sodium hydroxide solution and 0.5 c.c. of 30% hydrogen peroxide solution until a clear solution is obtained. The nitrate is then reduced to ammonia by addition of 10 c.c. of 30% sodium hydroxide solution and 0.25 g. of Devarda's alloy, and the ammonia is steam-distilled into *N*/70 hydrochloric acid, the excess of acid being titrated with standard sodium hydroxide solution. If a solution of nitrocellulose in an organic solvent is allowed partially to diffuse into the pure solvent, it is found that a separation into two fractions of different physical behaviour and of different nitrogen content has taken place.

H. F. GILLBE.

Deflocculation and detergency. CHAPIN.—See XII. Carbon disulphide in viscose manufacture. EGGERT.—See XXIII.

PATENTS.

Degreasing of raw wool. R. BRAUCKMEYER (G.P. 445,792, 28.5.25. Denm., 18.7.24. Addn. to G.P. 411,334; B., 1925, 842).—Wool dried until it contains only 2–3% of moisture is degreased with acetone and subsequently treated with water. L. A. COLES.

Lowering the viscosity of nitrocellulose in solutions by mechanical action. G. H. TOZIER, Assr. to EASTMAN KODAK Co. (U.S.P. 1,666,049, 10.4.28. Appl., 5.4.26).—The viscosities of solutions of nitrocellulose

are reduced to 80% of their original value by intensive mechanical agitation.

C. O. HARVEY.

Recovery of camphor [from celluloid]. A. FRIEDEN, Assr. to R. F. BACON and A. HIRSCH (U.S.P. 1,666,645, 17.4.28. Appl., 7.3.27).—Celluloid etc. is treated with an alkaline solution of such strength that the greater part of the nitrocellulose is not decomposed. On steam-distillation the camphor is removed.

B. FULLMAN.

Preparation of artificial silk of improved physical structure. T. IWASAKI and K. HAGIWARA (B.P. 288,655, 8.10.26).—See F.P. 621,543; B., 1928, 187.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Primary action of chromic acid on animal fibre. M. A. ILJINSKI and D. J. KODNER (J. Russ. Phys. Chem. Soc., 1928, 60, 193—207).—The action of chromic acid on wool fibre under various conditions was investigated. The fibre readily adsorbs the chromic acid in the cold, forming a bright yellow, stable complex. The amount adsorbed is proportional to the time of reaction and concentration of solution, and reaches a maximum at about 85% adsorption. The complex is, in every case, one of the basic fibre with dichromic acid, irrespective of the nature of the original chromium salt. The maximum amount taken up by the fibre is 0.09—0.1 g.-mol./100 g. of wool. Addition of sulphuric acid above a certain limit, as also rise of temperature, have no effect on the final adsorption. The dichromate complexes are not hydrolysed by water, like the sulphuric and hydrochloric acid complexes—both the latter are quantitatively displaced by addition of chromic acid. Silk fibre, likewise, forms dichromic acid complexes in the cold, and this property affords a means of dyeing woollen and silk fibre with lake colours and effecting an economy of more than 90% in the use of steam in mordanting. For colours which are easily oxidised, the dichromic acid complex can be reduced on the fibre by means of bisulphite.

M. ZVEGINTZOV.

Mordanting of basic dyes. M. MICHELS (Giorn. Chim. Ind. Appl., 1927, 9, 580—588).—The various theories as to the nature of the dyeing process are discussed, especially as regards basic dyes. The author's results confirm those of Caille (B., 1926, 355), and indicate that absorption of basic dyes by Celanese is due, not to the cellulose acetate, but to the impurities present. Basic dyes are fixed more or less well by all silicates, provided that these are sufficiently finely divided, the capacity of the semi-colloidal clay, fuller's earth, etc. in this direction being more marked than that of any other natural silicate; the fastness of the dye towards light and washing increases with the micellar dispersion of the silicate. Quartz, even when very finely powdered, exhibits no tinctorial affinity, and silicic acid fixes basic dyes only when amorphous, the age and mode of precipitation of the acid exerting a pronounced influence on the composition of the lake precipitated. The fact that colloidal silicic acid precipitates only those basic dyes which contain two benzene nuclei is regarded as due to the fact that the freshly precipitated acid is imperfectly colloidal and thus partly ionised,

carrying a negative electric charge. Maturation of the silicic acid is accompanied by saturation of the secondary valencies producing the positive electric charge, with formation of non-dissociable and non-crystallisable complexes. The latter are able to exert tertiary affinities, by means of two of which they combine with the dye. Silica precipitated in pulverulent forms absorbs basic dyes to a slight extent, this being an adsorption or surface phenomenon. Such dyes may, indeed, be purified by absorbing them from their impure solutions by means of silica powder and subsequently liberating them by treatment with pyridine. The trioxides of chromium, molybdenum, tungsten, and uranium combine with basic dyes, including those derived from triphenylmethane, the solubility and stability of the precipitates varying progressively throughout the homologous series with the degree of complexity of the oxide. The fixation of basic dyes by zinc sulphite is brought about by the formation with water of compounds such as $5\text{ZnSO}_3 \cdot 8\text{Zn}(\text{OH})_2$, these fixing the dyes by means of tertiary valencies. Zinc ferrocyanide and zinc thiocyanate also form basic dyes containing zinc salts. Cuprous thiocyanate, used in photography for the production of coloured films and diapositives, acts well only when it contains a certain proportion of alkali thiocyanate—that is, when complex salt is formed. Better results are obtained when the silver image is transformed into one of cupric ferrocyanide; dyeings on this salt exhibit moderate fastness against light, but less against washing. Cuprous ferrocyanide forms an excellent mordant for basic dyes. The four conditions for the stable fixation of basic dyes are that: (1) the mordant contains active groups, not in the state of ions, but as radicals forming part of a complex molecule; (2) the active groups are distributed in heterogeneous molecules—that is, those containing also radicals inert towards basic dyes; (3) the compounds containing active groups are not crystalline, so that the molecules do not assume reciprocally symmetrical positions; and (4) these compounds occur in as highly disperse a state of aggregation as possible.

T. H. POPE.

PATENTS.

Dyeing of artificial [viscose] silk. BRIT. DYE-STUFFS CORP., LTD., J. BADDILEY, P. CHORLEY, and R. BRIGHTMAN (B.P. 287,010, 8.9.26. Addn. to B.P. 281,410; B., 1928, 121).—Level dyeings on regenerated cellulose materials are obtained by the application of secondary disazo dyes made by coupling a diazotised aminoazo compound with *N*-substituted derivatives of 2-amino-8-naphtholsulphonic acids, such as phenyl- γ -acid, β -naphthyl-H-acid, β -naphthyl- γ -acid, *o*-anisyl- γ -acid, 2:4-dinitrophenyl- γ -acid, ethyl- γ -acid, benzoyl- γ -acid, acetyl- γ -acid, and 8-hydroxy-6-sulpho- β -naphthyl-glycine. The shades obtained are browns, navy-blue, violet to black.

C. HOLLINS.

Production of fast dyeings [on acetate silk, pelts, hairs, feathers, etc.]. A. G. BLOXAM. From I. G. FARBENIND. A.-G. (B.P. 287,651, 5.1.27. Addn. to B.P. 262,537; B., 1927, 104).—The goods are padded with a mixture of a coupling component, an arylamine, and a nitrite, and the bath is acidified

to develop the colour; the nitrite assists the absorption of the azo components. The two-bath process of the prior patent for dyeing acetate silk is extended to the dyeing of pelts, hairs, feathers, etc. Degreasing ("killing") and protective materials may be added to the dye-bath. In the printing process of the prior patent the arylamine may be omitted from the printing paste and applied afterwards, preferably with addition of an organic acid. In the case of *m*-diamines, the goods are padded with the diamine and printed with acid and nitrite. Twenty-one examples are given.

C. HOLLINS.

Dyeing of regenerated cellulose materials. BRIT. DYESTUFFS CORP., LTD., and J. BADDILEY (B.P. 287,609, 10.12.26. Addn. to B.P. 283,319; B., 1928, 189).—Viscose silk is dyed in level blue shades by means of secondary disazo dyes (other than those of the prior patent) of the type: sulphonated or carboxylated *p*-nitroarylamine \rightarrow middle component \rightarrow an aminonaphtholsulphonic acid (except 2:8-aminonaphtholsulphonic acid) or *N*-derivatives thereof. The nitro-group may be reduced to give developing dyes. The dye *p*-nitroaniline-*o*-sulphonic acid \rightarrow cresidine \rightarrow 1:8-aminonaphthol-4-sulphonic acid gives bright blue shades.

C. HOLLINS.

Dyeing of regenerated cellulose materials. BRIT. DYESTUFFS CORP., LTD., and J. BADDILEY (B.P. 287,214, 13.12.26).—Viscose silk is dyed in level brown-black to black shades with secondary disazo dyes made by coupling a diazotised nitroarylamine containing no sulphonic or carboxylic groups with a middle component, rediazotising, and coupling in alkaline medium with a sulphonated 1:8-aminonaphthol or an *N*-substituted derivative thereof. The nitro-group may be reduced. Examples are: *p*-nitroaniline \rightarrow α -naphthylamine-6(7)-sulphonic acid \rightarrow 1:8-aminonaphthol-4-sulphonic acid (dull blue); *p*-nitroaniline \rightarrow cresidine \rightarrow H-acid, reduced (blue-black). The reduced dyes may be diazotised on the fibre and developed, *e.g.*, with *m*-tolylenediamine.

C. HOLLINS.

Mordanting and dyeing of materials made with or containing cellulose derivatives. H. DREYFUS (B.P. 286,761 and 287,204, 3.12.26).—(A) Cellulose esters or ethers are mordanted with solutions of mordant metal salts (other than ferric salts) at concentrations of 15% or higher and are then dyed with mordant dyes. If temperatures above 75–80° are used, a protective agent is added to prevent delustring. Examples are alizarin on an aluminium mordant, Modern Heliotrope DH on a chrome mordant. Pattern effects are obtained in the usual manner by printing or stencilling, and by local application of a resist. (B) The mordant metals are best applied in the form of their aliphatic salts, especially salts of hydroxylated acids, *e.g.*, lactates, glycolates, citrates, acetates, in concentrations of 5% or higher. Mixed salts, such as aluminium sulphate-acetate, nitrate-acetate, etc., and basic salts may be used.

C. HOLLINS.

Dyeing of artificial silk. J. BADDILEY, P. CHORLEY, and C. BUTLER, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,667,524, 24.4.28. Appl., 2.4.27. U.K., 4.6.26).—See B.P. 276,450; B., 1927, 812.

Production of figured fabrics. G. HEBERLEIN, Assr. to HEBERLEIN PATENT CORP. (U.S.P. 1,667,892, 1.5.28. Appl., 27.7.25. Ger., 29.7.24).—See B.P. 237,909; B., 1926, 485.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Titrometric determination of calcium and magnesium carbonates in limestone. J. S. PIERCE, W. C. SETZER, and A. M. PETER (Ind. Eng. Chem., 1928, 20, 436–437).—The carbonate is dissolved in a known excess of standard acid, the solution boiled, and titrated back with standard alkali until neutral to bromothymol-blue. There is then added 1 c.c. of saturated alcoholic trinitrobenzene for each 10 c.c. of solution, and alkali is run in until the indicator shows a dark brick-red colour. A blank test correction to the same colour is run immediately, as the colour is not permanent. The result indicates completion of magnesium precipitation. Results of analyses are in good agreement with those obtained gravimetrically. Aluminium, if present, must be filtered off at neutrality, but iron does not interfere unless present in such quantity as to mask the colour.

C. IRWIN.

Determination of very small quantities of iodides. J. T. DUNN (Analyst, 1928, 53, 211–212).—The following method, which is a modification of Hunter's method for iodine in thyroid glands (B., 1910, 784) and Brubaker's for natural waters (B., 1926, 630), is satisfactory. About 50 g. of the sample are dissolved in 250 c.c. of water, and a few drops of sodium hypochlorite solution are added, followed by 1–5 c.c. of 40% phosphoric acid. The chlorine is driven off by boiling, 1 or 2 c.c. of 1% potassium iodide are added to the cooled solution, and the liberated iodine is titrated with about 0.005*N*-thio-sulphate.

D. G. HEWER.

Properties of high-calcium lime. RAY and MATHERS.—See IX.

PATENTS.

Production of high-percentage nitric acid. N. CARO and A. R. FRANK (B.P. 273,718, 28.6.27. Ger., 29.6.26).—Increased concentrations of ammonia and oxygen are made possible in the catalytic formation of nitric acid by a layer of cooling liquid circulating in front of or under the contact layer and extending over its whole width so closely that no travelling explosion wave can be formed. The gaseous mixture bubbles under pressure through this liquid (water, ammoniacal liquor, etc.) in such a way that heat radiated from the contact material is absorbed and liquid is evaporated into the mixture. Devices for cooling the walls and for absorbing radiation heat on the nitric oxide side of the contact layer are indicated.

W. G. CAREY.

Combustion of ammonia. H. PAULING (B.P. 279,511, 24.10.27. Ger., 22.10.26).—Uniform distribution of the gas current over a large cross-section in the catalytic combustion of ammonia with air is obtained by a series of diffuser chambers in the form of orifices in a thick plate of refractory material, narrow ribs being left between the orifices upon which the contact mass is supported. The diffuser element is set in operation

by heating with combustible gas and air which flows axially into each diffusion chamber; the combustible gas is then replaced by ammonia. W. G. CAREY.

Manufacture of hydrofluoric acid. W. J. HARSHAW and C. S. PARKE, Assrs. to HARSHAW, FULLER, & GOODWIN Co. (U.S.P. 1,665,588, 10.4.28. Appl., 19.2.25).—A fluoride in broken, fragmentary form (not ground) is heated with a suitable acid to liberate hydrofluoric acid, any sulphate coating on the fluoride fragments being removed to allow further action. W. G. CAREY.

Manufacture of silica gels. H. N. HOLMES and J. A. ANDERSON (U.S.P. 1,665,264, 10.4.28. Appl., 10.8.23).—A soluble silicate and a metallic salt are mixed in solution, and the resulting mixture of hydrated silica, soluble salt, and insoluble metallic oxide is dried in a manner to produce a rigid structure without material shrinkage; the oxide and salts are subsequently dissolved out. W. G. CAREY.

Wide-porous active silica. Active silica gels with fine capillary pores. I. G. FARBENIND. A.-G. (B.P. 263,198—9, 20.12.26. Ger., 19.12.25).—(A) Active silica gels with wide pores, particularly suitable for the absorption of gases and vapours at high partial pressures and saturated or almost saturated vapours, are prepared by washing a silica jelly, before or during drying, with such a solution as to impart to it p_H 7—10. *E.g.*, a jelly prepared by stirring 550 litres of sodium silicate solution, d 1.164, with 51 kg. of sulphuric acid, 46 kg. of ice, and 60 litres of water, and subsequently heating the mixture, is washed until it gives a red-violet coloration with cresol-red, preferably with water treated with alkalis to give the same coloration, after which it is dried in a current of air at 150—200°. (B) Gels with fine capillary pores, having a high absorptive capacity for gases and vapours at low partial pressures, are prepared by a process similar to that described in (A), except that the jelly is washed until it has p_H 2—5.5, *i.e.*, until it gives a yellow to blue-green coloration with bromocresol-green, water acidified with hydrochloric to give this coloration being used for the washing. L. A. COLES.

Manufacture of prepared calcium chloride. A. K. SMITH, Assr. to DOW CHEM. Co. (U.S.P. 1,660,053, 21.2.28. Appl., 12.10.25).—A saturated solution of calcium chloride is prepared at 190°, cooled below 175° under pressure, and allowed to solidify before it is powdered. H. ROYAL-DAWSON.

Continuous transformation of oxides into chlorides by exothermic reaction. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 274,048, 25.5.27).—Chlorine is introduced into a shaft furnace containing a mixture of the oxide with a suitable amount of an inert solid substance, *e.g.*, retort carbon, to absorb the excess heat of reaction. The inert material is of such granular size that the separation of the chloride can be brought about by screening. W. G. CAREY.

Production of complex fluorides. A. F. MEYERHOFER, Assee. of M. BUCHNER (B.P. 269,491, 2.3.27. Ger., 13.4.27).—A suitable gaseous fluoride, with admixture of neutral gas if necessary, is injected into a tall column of liquid containing a metal fluoride, a salt of the base the fluoride of which is required, an acid, and

a solvent. The influx of gas causes the liquid to overflow, and it is returned to the lower part of the column while the solid reaction products are conducted downwards into a conically widened chamber. The process may be conducted under pressure or *in vacuo* and assisted by heating or stirring or by baffles in the column.

W. G. CAREY.

Preparatory treatment of crude heavy spar. K. EBERS (B.P. 288,498, 25.10.27).—Heavy spar containing little or no lime and in which silica and compounds of iron and manganese are present yields a pure white barium sulphate if it is roasted, quenched in water without cooling, and treated with hydrochloric acid. W. G. CAREY.

Production of hydrated iron oxide from iron metal. J. WAGNER (B.P. 287,702, 8.3.27).—Scrap iron or steel free from rust is treated in an open tank with a continuous current of an aqueous solution containing up to 3% of sodium chloride and up to 1.5% of chlorides having catalytic properties, *e.g.*, ferric chloride, manganese chloride, etc., the liquid being continuously aerated with air to which about 10% of dust-free carbon dioxide has been added. The overflow from the top of the tank passes into a filter to remove the suspended hydrated iron oxide before the liquor is pumped back through an inlet at the lower end.

L. A. COLES.

Production of artificial fogs [from phosphorus]. A. V. JERNBERG (U.S.P. 1,665,267, 10.4.28. Appl., 14.7.26. Swed., 22.7.24).—A continuous supply of melted phosphorus subjected to fluid pressure is passed through a perforated device, and the finely-divided material so produced is delivered to the open air.

F. G. CLARKE.

Production of helium from natural gas. P. E. HAYNES, Assr. to LINDE AIR PRODUCTS Co. (U.S.P. 1,664,412, 3.4.28. Appl., 7.8.19).—Natural gas is compressed, cooled, and expanded to liquefy the greater portion of the hydrocarbons, the liquid is rectified to recover dissolved helium, and the mixed gases are further cooled without compression to liquefy progressively the other constituents, which are collected separately and utilised separately in progressively cooling the residual gas until it consists of practically pure helium.

A. R. POWELL.

Concentration or purification of caustic soda or other suitable material. C. F. HAMMOND, Assr. to W. SHACKLETON (U.S.P. 1,668,504, 1.5.28. Appl., 4.9.26. U.K., 5.9.25).—See B.P. 265,252; B., 1927, 251.

Separation of compounds [nitrates] of potassium from compounds [nitrates] of aluminium. H. J. FALCK, Assr. to NORSK HYDRO-ELEKTRISK KVAELSTOFABT. (U.S.P. 1,667,968, 1.5.28. Appl., 5.1.25. Norw., 27.2.24).—See B.P. 230,045; B., 1925, 758.

Production of alkaline-earth sulphides from alkaline-earth sulphates. T. LICHTENBERGER and K. FLOR (U.S.P. 1,667,423, 24.4.28. Appl., 4.3.26. Ger., 8.5.25).—See B.P. 251,942; B., 1927, 75.

Manufacture of arsenates of manganese. W. L. TANNER, Assr. to GRASSELLI CHEM. Co. (Re-issue 16,933, 10.4.28, of U.S.P. 1,591,795, 6.7.26).—See B., 1926, 743.

‡ **Catalytic combustion of ammonia-oxygen mixtures.** I. W. CEDERBERG (U.S.P. 1,666,958, 24.4.28. Appl., 8.12.24. Ger., 4.12.23).—See B.P. 225,821; B., 1925, 589.

Aluminium chloride from sludge (U.S.P. 1,665,406).—See II. **Formic acid** (U.S.P. 1,666,437).—See III. **Gases treated in electric arcs** (B.P. 263,859 and 288,056).—See XI. **Fumigating mixtures** (B.P. 271,514).—See XXIII.

VIII.—GLASS; CERAMICS.

Influence of iron oxide on the properties of glass. S. ENGLISH, H. W. HOWES, W. E. S. TURNER, and F. WINKS (J. Soc. Glass Tech., 1928, 12, 31–44).—In a series of glasses of the general molecular formula $6\text{SiO}_2 \cdot (2-x)\text{Na}_2\text{O} \cdot x\text{Fe}_2\text{O}_3 \cdot (\text{FeO})$, although the iron had been added to the batch as ferric oxide, there was a ratio of ferrous oxide to total iron, as ferric oxide, varying between 9.4% and 21.6%. Melting of the batches presented no difficulty over the range prepared (2.91–26.35% of iron oxides as Fe_2O_3), but the higher members gave a heavy boil in the later melting stages. Little attack by the glass was observed on the sillimanite pot used for melting. The density of the glasses increased continuously with increase of iron oxide content, and from the data the specific volume constants for ferrous and ferric oxides were deduced as 3.0 and 4.8, respectively. Annealing temperatures varied somewhat irregularly, but indicated that replacement of sodium oxide by ferric oxide increased, replacement by ferrous oxide decreased, the annealing point. There was, in general, a diminution of linear thermal expansion when soda was replaced by iron oxide, but the values were affected by the relative proportions of ferrous and ferric oxides present. In the thermal expansion curves, in addition to the upper and lower critical points, two change points occurred in the normal region, the first at 130–140°, the second at 240–250°, and at each there was an increase of the linear coefficient of 8–15%.

A. COUSEN.

Relationship between chemical composition and the resistance of glasses to the action of chemical reagents. II. **Glasses containing iron oxides.** V. DIMBLEBY and W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 52–57).—The chemical durability of the series of soda-iron oxide-silica glasses previously described (cf. preceding abstract) was determined and compared with that of other glasses previously tested (B., 1927, 12). Resistance to attack by water, hydrochloric acid, and sodium carbonate progressively increased as iron oxide replaced soda, but the resistance continuously decreased in the same circumstances when caustic soda was the solution employed.

A. COUSEN.

Casing of colourless by cobalt blue glass. I. **Thermal expansions.** W. E. S. TURNER and F. WINKS. II. **Setting rate.** S. ENGLISH and W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 57–74, 75–82).—I. Increasing quantities of cobalt oxide were incorporated in a series of soda-lime glasses up to 0.49%, and in a potash-lead oxide series up to 0.9%. Thermal expansions were determined in a specially designed

apparatus, details of which are given. In the soda-lime series the expansion was but little altered by the introduction of the colouring agent, whilst upper and lower annealing temperatures suffered a maximum variation of only about 5°. The total variation of expansion of the lead series was little more than 3%, but the cobalt affected the upper and lower annealing temperatures to a greater degree than with the lime glasses. Thermal expansion measurements of ruby, green, and blue commercial casing glasses, as compared with the parent glass, indicated that ruby deviated most in expansion rate in the normal region, whilst the green glass differed most in its upper and lower annealing temperatures. In practice, the ruby glass was most difficult to flash, proving the decisive factor to be coefficient of expansion.

II. Increasing amounts of cobalt caused the glasses to set more quickly in working. Viscosity measurements over the range 1400° to 700° in the case of the lime glasses gave lower values for the coloured members than for the parent glass throughout the range, whilst, below 750°, members with higher cobalt content set with greater rapidity than the colourless glass. Temperature measurements of the surface and interior, during cooling, indicated that the important factor inducing quicker setting in practice in the case of the blue glasses was the greater radiating power which they possessed. A. COUSEN.

Calculation of glass constants on the basis of recent investigations. E. ZSCHIMMER (J. Soc. Glass Tech., 1928, 12, 82–118).—The best method of expressing the composition of glass, in order to relate this to its physical properties, for commercial purposes, is that of oxide percentage, and for systematic comparison it is most convenient to set out the continuous succession of all possible percentage combinations in terms of the substituents (or permutants) used. Most recent work has followed the permutant method, which is described and explained. From this point of view, an examination of modern research on the physical properties of glass was commenced, properties first treated being the devitrification constants of soda-lime-silica glasses, the working viscosity of these glasses at high temperature, the effect of magnesia and alumina on the working properties of soda-lime-silica and of boric oxide on soda-silica glasses. A. COUSEN.

Ultra-violet light transmission of some colourless bottle glasses. D. STARKIE and W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 27–29).—Light transmission in the region 6500 to 2500 Å. was measured in the case of glasses from samples used as food containers. There was a very definite increase of transmission in the ultra-violet with decrease of iron oxide content of the glass. A. COUSEN.

Analysis of opal and alabaster glasses. J. D. CAUWOOD, J. H. DAVIDSON, and V. DIMBLEBY (J. Soc. Glass Tech., 1928, 12, 7–16).—An account of the methods of analysis adopted for the determination of constituents, particularly the opacifying agents, in opal and alabaster glasses. The agents considered were fluorine compounds, phosphates, tin oxide, and alkali sulphates and chlorides. A. COUSEN.

Analysis of opal glasses. W. SINGLETON and R. C. CHIRNSIDE (J. Soc. Glass Tech., 1928, 12, 18–24).

—A description of methods considered best, from the authors' experience, for the analysis of opal glasses.

A. COUSEN.

PATENTS.

Manufacture of glass. E. E. FISHER, Assr. to G. W. BATCHELL (U.S.P. 1,665,693—4, 10.4.28. Appl. [A] 5.4.27, [B] 6.4.27).—(A) A transparent glass, capable of decolorisation by selenium, is prepared from a batch containing a substantial proportion of barium sulphate. (B) A batch for glass-making contains, as a primary component, barium sulphide. A. COUSEN.

Oven or kiln for firing pottery and other ware. F. BENNION, H. J. PLANT, and J. B. CLARKE (B.P. 287,693, 22.11.27).—A circular kiln with a number of fire-mouths has a perforated tube or conduit in communication with a central hole in the oven floor leading to the chimney, the upper or inlet end of the tube extending nearly to the crown or is joined to it.

W. G. CAREY.

Ovens or kilns for firing tiles, bricks, etc. J. and J. W. MOSS (B.P. 288,383, 15.1.27).—Alternate fire-mouths in a circular oven communicate with up-flues between the wall of the kiln and a lining, and the flame, after striking the crown, is drawn downwards through a conduit in the centre of the floor and extending to about half the height of the oven. The other fire-mouths communicate with down-flues leading to openings in the bottom of the oven lining.

W. G. CAREY.

Manufacture of porous refractory bricks etc. I. SETTERBERG (B.P. 281,254, 8.11.27. Swed., 26.11.26).—Finely-powdered refractory clay, chamotte, kaolin, quartz, etc. is mixed with water and a substance which reacts with water to produce gas, *e.g.*, aluminium powder, calcium, zinc, calcium carbide, etc., and when evolution of gas has ceased the plastic dough is formed into blocks and burnt.

W. G. CAREY.

Tank furnaces for the manufacture of glass. GEN. ELECTRIC CO., LTD., W. W. WARREN, and J. F. HYSLOP (B.P. 288,668, 13.12.26).

IX.—BUILDING MATERIALS.

Effect of temperature and time of burning on the properties of high-calcium lime. K. W. RAY and F. C. MATHERS (Ind. Eng. Chem., 1928, 20, 415—419).—Samples of limestone were burned at temperatures varying from 900° to 1300° and for periods from 2 to 10 hrs. The resultant limes were tested for rates of slaking (time required to attain maximum temperature), rate of settling of the hydrates, plasticity, and time required for thickening of the slurry. The effect of increasing time of burning was small, but in the same direction as that of increase of temperature of burning. Increase of temperature reduced the rate of slaking and decreased the plasticity of the putty. A medium temperature of burning gave the slowest settling and greatest volume of settled suspension, also the slowest thickening of the slurry. The plasticity of the putty made by adding water to the dry hydrate is usually less than of that made direct from quicklime. In this case the most plastic putty is given by quicklimes made at 1100—1250°. In

general, a temperature of about 1100° seems most desirable considering all factors.

C. IRWIN.

PATENTS.

Rotary tubular kilns for treating cement etc. O. BOUZIN (B.P. 288,114, 8.12.27. Addn. to B.P. 266,939; B., 1927, 333).—The refractory lining of the kiln immediately behind the high-temperature zone is also provided with a ring-shaped projection, and the tubular clinker cooler is fitted internally with similar projections of refractory material in the upper portion of the cooler and of metal in the lower portion.

W. G. CAREY.

Manufacture of pavements. R. HADDAN. From WEST PROCESS PAVEMENT CO. (B.P. 287,799, 5.10.27).—A soft asphalt or flux is heated with pulverised hard asphalt and sand, with or without a filler.

L. A. COLES.

Treatment of rock asphalt. W. P. BENTLEY (U.S.P. 1,664,846, 3.4.28. Appl., 17.2.28).—A bituminous flux is added to the rock asphalt, previously reduced to a usable size, in amount insufficient in itself to fill the voids between the particles, but just sufficient to do so when added to the naturally contained bitumen in the rock.

A. B. MANNING.

Production of aqueous emulsions of bituminous material. H. MATTI (Swiss P. 119,219, 27.5.25).—The emulsions are prepared by adding hot water to mixtures containing bituminous material, a fatty oil which has been treated with concentrated sulphuric acid, and a sufficient quantity of an alkaline reagent to neutralise the sulphonic acids and free fatty acids.

L. A. COLES.

Manufacture of a bituminous product. E. HUTZENLAUB, Assr. to P. LECHLER (U.S.P. 1,665,881, 10.4.28. Appl., 26.6.26. Ger., 8.7.25).—A stable paste of a creamy consistency and non-miscible with water at ordinary temperatures consists of a tarry substance, water, and an emulsifying agent, the latter being insufficient in quantity to form an aqueous emulsion.

F. G. CLARKE.

Preservation of wood. J. HIMMELSBACH (F.P. 604,897, 20.10.25. Ger., 20.10.24).—Fresh wood, before impregnation with, *e.g.*, mercuric chloride solution, is thoroughly wetted with water and subjected for about 7 hrs. under reduced pressure to the action of the moist vapour of neutral solvents, *e.g.*, methyl alcohol or carbon tetrachloride, and formaldehyde, at a maximum temperature of 80—85°.

L. A. COLES.

Production on wood of a protective coating impervious to water. GEOR. HIMMELSBACH A.-G. (F.P. 600,290, 2.7.25. Ger., 15.1.25).—Wood is painted with a homogeneous mixture of bitumen or pitch with a solvent, *e.g.*, carbon tetrachloride.

L. A. COLES.

Mixing machines [for concrete]. J. M. VER MEHR (B.P. 288,365, 5.1.27).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Blast-furnace data and their correlation. E. C. EVANS and F. J. BAILEY (Iron and Steel Inst., May, 1928. Advance copy. 64 pp.).—From the operating

results of more than 120 blast furnaces, data in relation to output and fuel consumption have been correlated on a common basis. Other factors being equal, the greater the effective time of contact between the reducing gases and the ore before the hearth is reached the lower is the fuel consumption. Among the factors influencing the effective time of contact are (a) slow driving of the furnace, (b) preparation of the burden before charging, (c) methods of charging, (d) furnace design, and (e) quality of coke. Over a range of operation including a wide variation in furnace design, composition of ores, metals, and slags, the mean operating hearth temperatures are approximately constant, *i.e.*, metal $1450^{\circ} \pm 50^{\circ}$, slag $1500^{\circ} \pm 100^{\circ}$. The basic heat loss from the hearth zone is represented as necessitating the combustion of K/D lb. of carbon per sq. ft. of hearth area, where K is a constant and D the diameter of the hearth. By development an average equation for all the furnaces examined became $C_1 + C_2 = 500/D + 0.56I + 0.28S$, in which C_1 is the carbon gasified at tuyères, C_2 the sensible heat in blast in terms of carbon, I amount of iron (lb.) made, S the amount (lb.) of slag made, all figures being per sq. ft. of hearth area per hr.; in 80% of the cases examined coke consumptions calculated from these factors came within 10% of the actual amount. The amount of carbon required for external heat losses from the hearth is much greater than was generally thought, consequently slow driving is an uneconomical method of obtaining increased time of contact. The hearth losses are inversely proportional to the diameter of the hearth, and the heat consumption in the hearth is directly affected by the quality of the iron as represented by the content of silicon. Improved thermal efficiency, together with a possible increase in the effective rate of reaction between gases and ore, results from the use of dry blast. Constructional and operating details are given for 123 blast furnaces.

C. A. KING.

Comparison of the most important methods employed in the cleaning of blast-furnace gas. V. HARBORD (Iron and Steel Inst., May, 1928. Advance copy. 28 pp.).—Results obtained from comparative tests on dry- and wet-cleaning plants showed that the dust content of blast-furnace gas could be reduced to 0.4–0.5 g./m.³ by wet cleaners with spray towers only and by electrostatic cleaners. Spray towers followed by Theisen disintegrators and Halberg-Beth filtration plants gave the best results, dust being reduced to 0.02 or even 0.004 g./m.³ after a final spray for engine purposes. General considerations taking into account the maximum heat development and minimum erosion of refractories in relation to the cost of cleaning seem to indicate that a gas containing not more than 0.2 g./m.³ gives sufficiently good results. The electrostatic plants showed most favourable results on power and working costs, though the capital cost is high; the dust content of the cleaned gas was greater than with disintegrator washers, and the handling of dry dust presented some disadvantages. If due attention is given to the renewal of bags the Halberg-Beth cleaner gives a lower dust content, and its cost of operation is no more than that of the more efficient wet cleaners. Again high capital cost and dry dust are objections. The Kling-Weidlein

plant which depends on gas being passed through wire-wool mattresses is regarded only as a primary cleaner in conjunction with some other form, and its cost is too high for a first-stage cleaner.

C. A. KING.

Theory of blast-furnace smelting [of iron ores]. F. WÜST (Stahl u. Eisen, 1928, 48, 505–506).—To account for the considerable economy in fuel that follows from the use of a hot blast, the author outlines a new theory of the mechanism of the processes taking place during the blast-smelting of iron ores. For efficient working the depth of the oxidising zone just above the tuyères must be as small as possible; this may be effected by widening the furnace at this part, by the use of an oil spray, by means of a hot blast, or by substituting wood charcoal for coke. The thermal decomposition of carbon monoxide favours the reduction process and a hot blast increases this decomposition. The carbon liberated from this reaction effects the so-called direct reduction of iron, phosphorus, and silicon in the zone just above the tuyères, the indirect reduction higher up the furnace being brought about by hydrogen reduced by the carbon monoxide from the water vapour present. The water formed in the reduction is then again reduced to hydrogen by further quantities of carbon monoxide. The iron formed in the upper zones of the shaft takes up the greater part of the impurities found in the pig iron by cementation and not by direct reduction from the slag. The analysis of the flue gases or of gases taken from any part of the shaft is not sufficient to permit of any inferences being drawn as to the working of the blast furnace. Experimental details on which the theory is based are withheld for future publication.

A. R. POWELL.

New plant of the Appleby Iron Co., Ltd. -A. CROOKE and I. THOMSON (Iron and Steel Inst., May, 1928. Advance copy. 35 pp.).—A general survey of the plant, including blast furnaces, open-hearth furnaces, power and blowing plant, rolling, slabbing, and plate mills, basic slag plant, etc.

C. A. KING.

Blast-furnace practice in Natal. J. E. HOLGATE and R. R. F. WALTON (Iron and Steel Inst., May, 1928. Advance copy. 26 pp.).—A description of the materials and operation of the only blast furnace in S. Africa. One of the principal ores is a carbonate ore from Prestwick containing 5% of free carbon which renders the ore self-calcining. Northern Natal possesses the only extensive areas of coking coal in S. Africa, and a dolomite has been used from near Johannesburg (180 miles), replacing limestone previously obtained from Bechuana-land (400 miles). Costs of production are controlled largely by the high rates of carriage.

C. A. KING.

Influence of varying proportions of scrap and pig iron on the economical working of the open-hearth process [for the manufacture of steel]. G. BULLE (Stahl u. Eisen, 1928, 48, 329–338, 368–370).—Comparative data of the costs and working results of six steelworks using various ratios of pig iron to scrap in the Siemens-Martin furnace are tabulated and briefly discussed.

A. R. POWELL.

Peculiar graphite crystallisation in pig iron and cast iron. H. PINSL (Stahl u. Eisen, 1928, 48,

473—477).—Fifteen photomicrographs of peculiar forms in which graphite has crystallised in pig iron and cast iron are reproduced, and the factors which have probably influenced their formation are briefly discussed. The graphite-ferrite eutectic may frequently be found along veins of graphite segregations on the outer layers of very large castings. In pig iron with a high phosphorus content, the graphite veins often consist of numerous thin lamellæ of graphite separated by thin layers of iron phosphide, and sometimes a gradual transition from a vein of steadite to one of graphite may be observed. The mechanism of the formation of graphite nuclei and the structure of temper carbon are briefly discussed. A. R. POWELL.

Twin-like crystals in annealed α -iron. H. O'NEILL (Iron and Steel Inst., May, 1928. Advance copy. 9 pp.).—A metallographic examination has been made of certain small ferrite grains embedded in a large single crystal of iron prepared by the method of straining and annealing. Pressure figures, etch pits, and the form of the grain boundaries indicated that the grains were twins (relative to the large crystal), the twins being bounded by (112) planes, the twinning being of the fluorite type. They are regarded as true annealing twins produced by cold-work and recrystallisation.

W. HUME-ROTHERY.

Rate of dissolution of graphite in molten iron-carbon alloys. F. SAUERWALD and A. KORENY (Stahl u. Eisen, 1928, 48, 537—540).—The rate of dissolution of graphite in cast iron has been determined at 1255° and at 1350°. In both cases the rate approached closely to that calculated from the formula of Noyes, Withney, and Nernst ("Theoretical Chemistry," Stuttgart, 1926). It is proportional to the degree of saturation of the molten metal, the contour of the graphite particles, the temperature, and the time of contact. The amount of graphite dissolved in a given time increases rapidly with the temperature, and that dissolved at a given temperature increases at first rapidly with the time, then more slowly, the curve being hyperbolic in form.

A. R. POWELL.

Graphite in cast iron and its influence on the tensile strength. P. BARDENHEUER and K. L. ZEYEN (Mitt. K.-W.-Inst. Eisenforsch., 1928, 10, 23—53; Stahl u. Eisen, 1928, 48, 515—519).—The higher the temperature at which cast iron is heated the smaller is the amount of graphite which separates on cooling; cast iron even with a high silicon content has a white fracture after heating above 1500°. At very high temperatures (e.g., 1700—1800°) air or occluded gases and sometimes the walls of the crucible cause the separation of graphite nuclei, and the metal solidifies wholly or partly as grey cast iron (cf. Pivovarsky, B., 1925, 805; Hanemann, B., 1927, 678). The tensile strength of cast iron is very appreciably increased by casting in chill moulds and annealing at 850—900° for 6 hrs. owing to the finer state of subdivision of the graphite compared with that in sand castings. The ground mass in sand castings is chiefly pearlite, and that in annealed chill castings chiefly ferrite; hence it appears that the state of subdivision of the graphite is the factor which determines the mechanical properties of cast iron, and that the nature

of the ground mass has little influence. Numerous photomicrographs of the structure of cast iron heated at temperatures between 1200° and 1800° and cooled at varying rates are included.

A. R. POWELL.

Influence of nickel on iron-carbon-silicon alloys containing phosphorus. A. B. EVEREST and D. HANSON (Iron and Steel Inst., May, 1928. Advance copy. 23 pp.; cf. B., 1927, 782).—The influence of up to 3% of nickel on synthetic iron alloys containing 3.5% C, 1.2 and 2.5% Si, and over a range 0.2 to 1.2% P has been investigated; the experiments were afterwards repeated with a commercial refined iron containing 3.05% C and 1.87% Si. In all cases it was found that phosphorus increases the tendency of the iron to chill; this chilling effect may be counteracted by the addition of small quantities of nickel, and is less pronounced in the high-silicon irons. Phosphorus increases the hardness of the iron; in the absence of chill, nickel produces its normal hardening action. Phosphorus impairs the machinability of the iron. Both nickel and phosphorus have a refining effect on the graphite in the iron, but with a high phosphorus content the effect of nickel is small and inappreciable when 1.2% P is present.

M. E. NOTTAGE.

Annealing transformer sheet [iron]. M. VON MOOS, W. OERTEL, and R. SCHERER (Stahl u. Eisen, 1928, 48, 477—485).—Recrystallisation tests on 4% silicon-iron for transformer sheets showed that the presence of oxygen has almost as great an effect on the wattage losses as the presence of carbon; it prevents the formation of an even, regularly oriented crystal structure during annealing and restrains the grain growth. The harmful action of carbon and oxygen in transformer sheets may be overcome by annealing in hydrogen, which reduces the oxides and produces a sheet which, in the absence of other harmful constituents, has a very low wattage loss. A period of 1 hr. at 900—1000° suffices for most sheets; longer annealing at 1000° produces slightly higher losses. A. R. POWELL.

Resistance of over-stressed wrought irons and carbon steels to salt-water corrosion. J. N. FRIEND (Iron and Steel Inst., May, 1928. Advance copy. 12 pp.).—Bars of wrought iron and carbon steels were deformed by stretching, twisting, or compression, and were then immersed for one or two years in salt water, made by dissolving rock salt in water to give a solution resembling sea water. To examine the effect of surface conditions the bars were immersed both in the natural state after deformation, and also after machining to shape. Under these conditions the corrodibility of wrought iron is not increased by stretching even to rupture, by twisting, or by compression up to 30%. The corrodibility of carbon steels (0.025—1.35% C) is not enhanced by stretching even to rupture. Torsion and compression do not affect the corrodibility of 0.11% carbon steel, but with 0.20% carbon steel the corrodibility is slightly increased. Wrought irons and carbon steels (0.025—0.32% C) deformed by stretching, and then tested in the natural condition without machining the surface, were in nearly every case less corroded than the same material in the unstressed state; in the case of the 0.11% carbon steel the loss on weight of the

stretched bar was 20.4% less than that of the unstressed bar.

W. HUME-ROTHERY.

Properties of materials at high temperatures.

III. "Creep" of Armco iron. H. J. TAPSELL (Dept. Sci. Ind. Res., Eng. Res., Spec. Rep. No. 6, 1928. 11 pp.; cf. Tapsell and Clenshaw, B., 1927, 525).—"Creep" tests made on Armco iron at 136° under a load of 25.7 tons/in.² showed that creep had practically ceased in 22 days. Stress-strain relations of specimens which had been creep-tested at 150°, 237°, 325°, and 390° indicated that plasticity progressively decreased and the new limit of proportionality acquired by the material was at all temperatures much greater than the value of the original material. It is considered that a hardening process occurs during creep similar to that taking place in the neighbourhood of the slip planes during a fatigue test.

C. A. KING.

Fatigue-resisting properties of 0.17% carbon steel at different temperatures and at different mean tensile stresses. H. J. TAPSELL (Iron and Steel Inst., May, 1928. Advance copy. 13 pp.; cf. B., 1927, 525).—Fatigue limits at different mean tensile stresses and at temperatures up to 500° were determined in a Haigh machine under direct pulsating stresses applied at the rate of 2400 cycles/min. for 10⁷ cycles. For practical use, the results obtained must be correlated with a knowledge of the yield-points and limiting creep stresses at different temperatures. It is shown that the endurance of the steel above 300° is wholly dependent on the rate of alternations applied; at air temperature the rate has little, if any, effect. Large ranges of stresses can be applied at all temperatures. The superior limit of ranges of stress applied at 2400 cycles/min. for 10⁷ cycles can exceed the ordinary ultimate strength values. The "practical" fatigue limits under different mean tensile stresses do not appreciably alter for temperatures up to 200° or 250°, and they are dependent on the yield-point of the material at the specified temperature. Above about 200° the practical fatigue limits are dependent on a suitable proof stress.

M. E. NOTTAGE.

Rapid normalising of overstrained steel. W. E. WOODWARD (Iron and Steel Inst., May, 1928. Advance copy. 10 pp.).—The elastic properties and resistance to fatigue of specimens of mild steel which have been seriously overstrained can be rapidly and completely restored by the passage of a direct current of about 600 amp. through the material so as to heat it above the upper critical point. Normalisation by this method produces remarkably uniform results. An alternating current of about 2000 amp. at 6 volts can also be used, but the results are less uniform. The results obtained by both methods compare very favourably with those obtained by the ordinary process of normalising.

M. E. NOTTAGE.

Heat-resisting steels. II. Mechanical properties. W. H. HATFIELD (Iron and Steel Inst., May, 1928. Advance copy. 22 pp.; cf. B., 1927, 445).—To compare the effects of the introduction of special elements into steel on its mechanical strength at high temperatures, tensile tests were made at 800°. For

ordinary steels, 7 and 8 tons/in.² indicates the limiting maximum stress value at 800°; high-speed tool steels give slightly higher values. Silicon-chromium heat-resisting steels are already softened at 800°. With steels containing 0.07 and 0.09% C, the presence of 14 and 18% of chromium, respectively, has no influence on the resistance to softening at high temperatures; increasing the carbon raises the strength. The addition of nickel has no influence even with a high carbon content, but nickel and chromium together increases the strength to a moderate extent. The introduction of tungsten results in a marked increase in strength when added together with nickel and chromium; 14% of chromium is about the requisite amount, but the amount of nickel may vary within wide limits. The addition of molybdenum, and to a lesser degree silicon, strengthens the iron-nickel-chromium steels at high temperatures. Carbon plays an important part, and there is an optimum percentage for the maximum strength in each combination in the alloys. It is probable that these results would be modified by varying the heat-treatment accorded to the material. The author discusses the phenomena accompanying deformation and rupture at high temperatures, and the effect of added elements on the steel from the point of view of the space lattice.

M. E. NOTTAGE.

Structure of the iron-chromium-carbon system.

A. WESTGREN, G. PHRAGMÉN, and T. NEGRESKO (Iron and Steel Inst., May, 1928. Advance copy. 18 pp.).—X-Ray analysis shows that the iron-chromium alloys at ordinary temperatures consist of an unbroken series of solid solutions, the size of the unit cube increasing regularly from $a = 2.861 \text{ \AA.}$ for pure iron to 2.878 \AA. for chromium. In the ternary iron-chromium-carbon system the following phases are met with: (1) α -Solid solution (body-centred cubic structure). (2) γ -Solid solution (face-centred cubic structure). (3) Cementite (Fe_3C) derived from ordinary cementite Fe_3C by the substitution of chromium for iron; the chromium content may rise to rather more than 15%. The lattice dimensions of cementite decrease slightly as the iron is replaced by chromium. (4) Cubic chromium carbide, probably $(\text{Cr},\text{Fe})_4\text{C}$ derived from Cr_4C (cf. Westgren and Phragmén, B., 1922, 418; 1924, 518) by the substitution of iron for chromium up to a maximum of about 25%, the lattice parameter decreasing with rising percentage of iron. (5) Trigonal chromium carbide $(\text{Cr},\text{Fe})_3\text{C}_2$, derived similarly from Cr_3C_2 (cf. Westgren and Phragmén, *loc. cit.*), in which the iron content may rise to 55%, the lattice parameter decreasing with rising percentage of iron. (6) Orthorhombic chromium carbide, $(\text{Cr},\text{Fe})_3\text{C}_2$, in which only a few per cent. of chromium may be substituted by iron. None of the above phases is a true double carbide, *i.e.*, a carbide requiring both chromium and iron atoms for its formation. In an annealed chromium steel such as is used for ball bearings almost all the chromium is in the cementite, and the constituent commonly called "double carbide" is simply ternary cementite in large crystals. In stainless steel the carbide is cubic chromium carbide saturated with iron. The equilibrium diagram of the ternary system is discussed in the light of these results.

W. HUME-ROTHERY.

Heterogeneity of steel ingots. Report II (Iron and Steel Inst., May, 1928. Advance copy. 147 pp.; cf. B., 1926, 490, 883).—The design of moulds and the effect of inclusions in steel on the heterogeneity of ingots are discussed, and macrographic and analytical representations of segregation in ingots of nickel, nickel-chromium, and nickel-chromium-molybdenum steels are given. Interim reports on the density and viscosity of molten steel, and on the freezing and melting ranges of various types of steel are included. C. A. KING.

Chromium steel rails. T. SWINDEN and P. H. JOHNSON (Iron and Steel Inst., May, 1928. Advance copy. 14 pp.).—The advisability of obtaining increased resistance of rails to wear by abrasion by increasing the carbon range and reducing the maximum manganese permitted is considered not beyond doubt, and from results of trials of steels containing up to 2% Cr, a chromium content of approximately 1% was adopted as being most satisfactory, other constituents being about 0.5% C, 0.8% Mn, 0.22% Si, 0.03 S, and 0.04% P. The effect of increasing the carbon content from 0.51% to 0.61% was to increase the maximum stress from 59.2 to 65.12 tons/in.², and the Brinell hardness value from 269 to 293. As rolled, the chromium steel shows a dense sorbitic pearlite matrix with a fine broken network of ferrite, the effect of the chromium addition being to reduce the proportion of the soft ferrite constituent and to emulsify the carbide, both conditions tending to contribute to the greater hardness and toughness of the chromium steel. C. A. KING.

Application of metallography in improving the quality [of iron and steel]. H. MEYER (Stahl u. Eisen, 1928, 48, 506—515).—Many examples are given with photomicrographs of various faults which can be found in iron and steel castings and forgings by the use of the microscope and by mechanical testing. The effect of transverse crystallisation, of segregation, and of blow-holes on the properties of the metal is discussed briefly with reference to certain specific examples.

A. R. POWELL.

Properties of nickel steels, with special reference to the influence of manganese. J. A. JONES (Iron and Steel Inst., May, 1928. Advance copy. 36 pp.).—The transformation points, mechanical properties, and microstructures of nickel steels containing 0.2—0.55% C and 3—12% Ni have been examined with special reference to the effects of manganese and chromium. In nickel steels the A_{cl} point is lowered by about 10° for each 1% of nickel, whilst with from 3 to 5% of nickel the A_{rl} point is lowered by from 25° to 30° for each 1% of nickel, the exact value depending on the manganese and carbon contents; the rate of cooling used was 4°/min. Consequently with low manganese content there is no advantage in increasing the nickel beyond 6% on account of the limitation on the tempering temperature imposed by the lowering of A_{cl}. Little effect on the critical ranges is produced by less than 0.8% of manganese; an increase in manganese content from 0.4 to 0.8% in steels with 4% Ni lowers the A_{cl} and A_{rl} by 3° and 4°, respectively, the rate of cooling being 4°/min. Chromium produces a decided rise in A_{cl}, but has little effect on A_{c3}, A_{r3}, or A_{rl}. On increasing the

manganese content of steels containing 3—4% Ni, a sudden improvement in mechanical properties takes place at a definite manganese content which increases with the size of the section treated; in no case should less than 0.5% of manganese be present. The mass effect met with in quenching these alloys has been studied in detail.

W. HUME-ROTHERY.

Effect of silicon on tungsten magnet steel. J. SWAN (Iron and Steel Inst., May, 1928. Advance copy. 9 pp.).—The "spoiling" or loss of coercive force on annealing above the A_c point of a tungsten magnet steel of normal composition is due to the gradual formation and segregation of free tungsten carbide, which is only slightly soluble in austenite at the ordinary hardening temperatures, so that the steel loses in available carbon and tungsten content. By increasing the silicon content to 0.25% or above this segregation can be retarded, but not prevented, as is shown by the fact that loss of coercive force still occurs on annealing. Silicon, up to 0.5%, has, if anything, a slightly beneficial effect on the magnetic properties of the steel; 1.0% of silicon reduces the loss of coercive force on annealing, but has a harmful effect on the general magnetic properties. It is probable that less loss due to ageing is shown by steels with a high silicon content.

M. E. NOTTAGE.

Iodometric determination of vanadium in special steels and in ferrovanadium. K. ROESCH and W. WERZ (Z. anal. Chem., 1928, 73, 352—355).—For the determination of vanadium in a special steel, 3—5 g. are dissolved in 30—50 c.c. of hydrochloric acid and sufficient nitric acid to effect complete oxidation, the solution is evaporated to 25 c.c., the tungstic acid and silica are filtered off, and the iron is removed by the ether-extraction method. The aqueous layer is evaporated to dryness and the residue dissolved in 20 c.c. of 1:1 nitric acid. Manganese sulphate is added, followed by ammonia to precipitate manganese vanadate and chromate. The precipitate is collected, washed, dried, ignited in an iron crucible, and fused for 10 min. with 3—4 g. of sodium hydroxide in an atmosphere of hydrogen. After cooling, the sodium vanadate is extracted with hot water, the insoluble chromic oxide filtered off, and the filtrate acidified with 75 c.c. of phosphoric acid (*d* 1.7). To ensure that all the vanadium is present as vanadate the solution is boiled for 15 min. with 0.5 g. of ammonium persulphate. The cold solution is treated with potassium iodide and the liberated iodine titrated with thiosulphate (cf. Heczko, A., 1926, 1020). For the analysis of ferrovanadium, 1 g. is fused in a nickel crucible with 10 g. of sodium hydroxide, the product leached with water, and an aliquot part of the filtrate treated with phosphoric acid and potassium iodide as above.

A. R. POWELL.

Rapid determination of gases in metals, especially oxygen in steel. W. HESSENBRUCH and P. OBERHOFFER (Arch. Eisenhüttenw., 1927—8, 1, 583—603; Stahl u. Eisen, 1928, 48, 486—487).—The efficient removal of occluded and combined gases from steel requires a temperature of 1500—1600° and a high vacuum, and the only suitable container for the specimen during the test is a graphite crucible which has previously

been degassed at 1500–1600° *in vacuo*. The apparatus used comprises a high-frequency induction furnace, a pump for producing a high vacuum, and the usual gas-analysis apparatus for the determination of carbon monoxide, carbon dioxide, hydrogen, and nitrogen. The graphite crucible is placed inside a magnesia crucible which is inside a closed cylindrical, silica container connected to the vacuum pump and placed inside the coil of the furnace. The specimen for analysis should consist of a solid block of the metal and not of turnings. Usually 45–60 min. at 1600° suffices to reduce all oxides and expel occluded gases. When more than 0.05% S is present in the metal errors may be caused by the formation of sulphur dioxide or hydrogen sulphide in the test.

A. R. POWELL.

Relative corrodibilities of ferrous and non-ferrous metals and alloys. I. Results of four years' exposure in the Bristol Channel. J. N. FRIEND (Inst. Metals, March, 1928. Advance copy. 23 pp.).—Bars of ferrous and non-ferrous metals were exposed to sea action in the Bristol Channel for four years, under conditions in which they were totally immersed for slightly more than 93.5% of the total time. Results are given for 14 non-ferrous, and 4 ferrous bars, losses in weight, depths of pitting, reduction in diameter, and fall in tensile strength having been measured. The detailed behaviour of each bar is described. Of brasses, both Muntz metal and Naval brass underwent dezincification, but not Nergandin brass or screw metal; these last two brasses corroded very uniformly with no local pitting. Aluminium was badly pitted, some holes penetrating to a depth of 5.8 mm. in a bar of 2.87 cm. diam. Taking pitting, loss in tensile strength, and total loss in weight into account, the non-ferrous metals are arranged in the following order of merit: English common ingot tin, high-grade pure tin, commercial nickel, antimonial lead, commercial soft lead, screw metal (Cu 60.02%, Zn 38.61%, Pb 1.37%, As < 0.05%), nickel-copper (1.75 Ni), arsenical copper (0.45% As), ordinary copper, Nergandin brass (Cu 70.05%, Zn 27.93%, Pb 2.02%), Naval brass, Muntz metal, commercial zinc, and commercial aluminium.

W. HUME-ROTHERY.

Annealing of non-ferrous metals in the electric furnace. R. M. KEENEY (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 10 pp.).—In a discussion of the relative merits of oil, gas, and electric furnaces for the annealing of non-ferrous metals, it is emphasised that the over-all cost of the finished product should be the deciding factor. The cost of thermal energy alone does not give a true basis of comparison, since items such as cost of labour, maintenance, rejects, pickling, etc. may vary greatly according to the source of heat adopted. Data are given for the electric furnace annealing of brass and copper tubing and nickel-silver stampings at Connecticut, and the saving in over-all costs of these processes as compared with oil-fired furnaces is estimated. It is concluded that the continued use of oil-fired furnaces in rolling mills is largely due to failure to consider economies in over-all cost which the introduction of electric furnaces might produce in this field.

H. J. T. ELLINGHAM.

Changes of a nickel-copper alloy in the vapour of superheated water at about 350–400°. J. F. SAFFY

(Compt. rend., 1928, 186, 1116–1118).—A plate 3 mm. thick of an alloy of nickel (68.6%), copper (28.9%), manganese (1.6%), and carbon (0.2%), which was originally pliable, broke with a deep crack after 2 weeks in steam at 500° when bent at an angle of 180°. After 7 weeks at 400°, it broke when folded to 90–120°, and after 8 weeks at 350°, cracks of varying depths appeared when it was bent to 180°. In some cases traces of a white oxide were noted, but there was little or no gain in weight.

J. GRANT.

Behaviour of metals and alloys during hot-forging. W. L. KENT (Inst. Metals, March, 1928. Advance copy. 18 pp.).—Small cylindrical specimens of tin, lead, zinc, aluminium, copper, and brass (both 70:30 and 60:40, with and without small amounts of lead) were forged with a standard blow of 50 ft.-lb. at temperatures up to the m.p. The deformation produced, and the Brinell hardness of the samples before and after deformation, were measured. When a material is worked at high temperature it strain-hardens in much the same way as, but to a slightly less extent than, at normal temperatures; the decrease is, however, small. With copper at 600°, recrystallisation after deformation is practically instantaneous and continuous forging is possible, but with aluminium residual hardness remains even after forging at 650°, although here the actual softness of the metal aids hot-forging. The forgeability of 70:30 brass increased but little up to 750°, but that of 60:40 brass increased steadily from 400° to 650°, and then rapidly to the m.p. The presence of lead caused cracking in brasses forged from 350° upwards, but in 60:40 brass the cracks disappeared at 700° and the lead was beneficial. In general, both forging and Izod notched-bar impact tests are necessary to give a true measurement of malleability, neither test alone being satisfactory.

W. HUME-ROTHERY.

Deterioration of lead cable sheathing by cracking, and its prevention. S. BECKINSALE and H. WATERHOUSE (Inst. Metals, March, 1928. Advance copy. 25 pp.).—The intercrystalline failure sometimes found in lead cable sheathing has been investigated by laboratory experiments and by the examination of samples which have failed in practice. The failure is a fatigue phenomenon, and test pieces submitted to alternating stresses fail by the development of intercrystalline cracks identical with those occurring in cable sheathing, but similar cracks could not be obtained by any other methods. In agreement with this, failures in practice usually occur only in places where the lead cable is subjected to vibration. The probability of failure can be greatly reduced by using binary or ternary alloys of lead with small quantities of tin, antimony, or cadmium in which the fatigue limit is much higher than in pure lead; alloys containing 0.25% Cd and 1.5% Sn, or 0.25% Cd and 0.5% Sb, are particularly suitable. No evidence could be found for any allotropic transformation of lead between –75° and +350°. The effects of simultaneous corrosion and tensile stress were also investigated, whilst other possible causes of intercrystalline fracture in lead are shown to be untenable.

W. HUME-ROTHERY.

Alloys of zirconium. I. T. E. ALLIBONE and C. SYKES (Inst. Metals, March, 1928. Advance copy.

14 pp.).—The microstructures of the alloy systems copper-zirconium to 35% Zr, nickel-zirconium to 55% Zr, and iron-zirconium to 30% Zr have been investigated, the alloys being prepared in a vacuum high-frequency induction furnace which is described. Copper dissolves only about 0.2% of zirconium in solid solution; the alloys contain a compound Cu_3Zr (m.p. above 1000°), which forms a eutectic with the copper solid solution at 964° and 12.5% Zr. The solid-solubility of zirconium in nickel is less than 0.5% at room temperature; the alloys contain two compounds, Ni_4Zr and Ni_3Zr , the former forming a eutectic with the nickel solid solution at 16% Zr. The addition of zirconium to iron lowers the Ar3 change point, but does not affect the Ar2 point. Iron dissolves about 0.3% of zirconium in the solid state at room temperatures; a eutectic alloy is formed at 15% Zr, but the second constituent of this eutectic has not been isolated. The hardness, tensile strength, and ductility of some of the alloys were investigated.

W. HUME-ROTHERY.

Speiss and the metals of the platinum group.

H. RUSDEN and J. HENDERSON (J. Chem. Met. Soc. S. Africa, 1928, 28, 181—196).—In recovering the platinum metals from certain concentrates on the Rand, the material is smelted to a speiss in which all the platinum metals are collected. A typical speiss contains 19.5% As, 56% Fe, 8% Cu, 8% Ni, 3.3% Pb, and 4% S, with about 100 oz./ton of platinum metals. By smelting this speiss in a cupel bed with 8 pts. of litharge, 1.36 pts. of sodium carbonate, and 0.7 pt. of sand, the platinum metals were obtained in a lead bottom weighing 4 times as much as the original speiss. This could be cupelled to about one third the weight of the speiss treated without serious loss of the precious metals, and by dissolution of the remaining lead in nitric acid a residue containing 13% of platinum metals was obtained. The amount of lead required in the above treatment was considerably reduced by roasting the speiss previous to smelting. Direct electrolysis of the speiss in 5% sulphuric acid yielded an anode slime weighing 26% of the original speiss; this slime lost 26% of its weight on roasting and, after fusion with iron and a borosilicate slag, gave a new speiss weighing 25% of the slime, together with a matte and slag practically free from the platinum metals. Electrolysis of the new speiss yielded 90% of the platinum metals in the anode slime, which contained 3.37% of these metals. Instead of smelting the first slimes to obtain a new speiss, the platinum metals could also be separated by roasting the slimes and smelting with litharge to obtain a lead bottom for acid treatment. All the above methods gave extractions well below 100%.

A. R. POWELL.

Ball hardness and cold-working of soft metals and eutectics. F. HARGREAVES (Inst. Metals, March, 1928. Advance copy. 27 pp.).—The relation between the diameter of the impression in the Brinell hardness test and the time of application of the load has been investigated for pitch, vulcanite, and a number of soft metals and eutectics in cast, annealed, or cold-worked states. The relation may be expressed by the general equation $d = ct^s$, where d is the diameter of the impression, t the time of application of the load, and c

and s are constants depending on the material, the diameter of the ball, and the applied load. Generally speaking the harder the metal the smaller are both c and s . The factor s is probably a measure of the rate of spontaneous annealing, and for a given metal it increases when the material is tested in the cold-worked state. The factor c for a given metal is almost the same for cast, worked, or annealed specimens. For soft metals the exact temperature at which the test is carried out is of great importance, the diameter of the impression being affected by as much as 0.005 mm. per 1°F . The significance of the ordinary Brinell numbers is discussed.

W. HUME-ROTHERY.

Recovery and sinking-in or piling-up of material in the Brinell test, and effects of these factors on correlation of the Brinell with other hardness tests. A. L. NORBURY and T. SAMUEL (Iron and Steel Inst., May, 1928. Advance copy. 15 pp.).—Measurements have been made of the "sinking-in" or "piling-up," and "flattening" and elastic recovery of 10 mm.-ball Brinell impressions on vulcanite and a number of metals in different conditions. If expressed as a percentage of the measured depth of impression, the sinking-in or piling-up of the material round Brinell impressions has a constant value for a given material, irrespective of the size of the impression. In cast materials the sinking-in, if expressed as above, may be as much as 30%, whilst in cold-worked materials the piling-up may reach this value. Hence depth indicators measuring the depth from the original surface may give differences of $\pm 30\%$ if the hardness be calculated from the indicated depths instead of from the diameter. The effects of these phenomena on the correlation of Brinell tests with scleroscope, pendulum, and cone indentation tests are discussed.

W. HUME-ROTHERY.

Adsorption processes in flotation. K. KELLERMANN and E. PEETZ (Kolloid-Z., 1928, 44, 296—308).—The adsorption of caprylic (octoic) acid by quartz and by galena was studied by measurements of the refractive index, electrical potential, electrical conductivity, and surface tension. Examination of the system quartz-octoic acid showed that, although quartz does not act as an adsorbent, a portion of it goes into colloidal solution under the peptising influence of octoic acid, and in this state it acts as an adsorbent. Galena is attacked by octoic acid, and the measurements were carried out in presence of a solution saturated with respect to lead octoate. Galena adsorbs octoic acid well, although the dried material does so poorly. The constants of the adsorption isotherms have been calculated from the experimental data, and also the molecular thickness of the adsorption layers. Under the experimental conditions, galena is readily floated, whilst quartz is not; the parallel between flotation and adsorption is discussed in regard to flotation practice.

E. S. HEDGES.

Aluminium and its alloys. BUSCHLINGER.—See I. **Coke for steel making.** WHEELER.—See II. **Magnet steels.** SANFORD. **Chromium.** LUKENS. **Nickel electrotyping.** WINKLER and BLUM.—See XI.

PATENTS.

Blast furnace. W. P. POWER and E. LEWIS, ASSTS. to BROKEN HILL PROPRIETARY CO., LTD. (U.S.P. 1,664,832,

3.4.28. Appl., 20.1.27. Austral., 9.12.26).—The upper part of a blast furnace is lined with refractory material which is supported against the shell independently of the lining in the lower part of the furnace. A. R. POWELL.

Cupola. A. H. COPLAN (B.P. 288,072, 18.7.27).—The refractory lining of a cupola is replaced by an inner casing of heat-resisting steel having ribs in helical formation on its outer surface in order to impart a cyclonic motion to a current of air passing between the inner and outer casings. The heated air is then conveyed to the air box and tapering tuyères of the cupola.

C. A. KING.

Open-hearth furnace. G. L. DANFORTH, JUN., Assr. to OPEN HEARTH COMBUSTION Co. (U.S.P. 1,665,086, 3.4.28. Appl., 13.7.22).—The furnace is provided with slag pockets which are connected by means of passages to the regenerative chambers and to the furnace ports. Vertical dampers are provided to close certain of the passages between the regenerative chambers and the slag pockets and to place them in direct communication with the furnace ports.

A. R. POWELL.

Tilting-hearth tray furnace. A. D. KEANE, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,664,749, 3.4.28. Appl., 28.3.27).—The furnace comprises an annular chamber, means for moving a number of trays through the furnace, and means for tilting the trays in the furnace and for returning them to their normal position.

A. R. POWELL.

Working-up ores and metallurgical products of various kinds containing volatilisable metals. F. KRUPP GRUSONWERK A.-G. (B.P. 287,745, 24.5.27. Addn. to B.P. 252,679; B., 1927, 583).—Additions of materials, *e.g.*, limestone, sand, intended for stiffening metallurgical substances in a refining furnace are introduced wholly or in part into the reaction zone and there mixed with the charge, enabling a higher temperature to be used. Other materials, *e.g.*, potassium nitrate or fine-grained ore, may be blown in together with additional fuel if desired.

C. A. KING.

Metallurgical briquette and process of using it. W. B. RUNYAN (U.S.P. 1,666,312, 17.4.28. Appl., 31.3.21).—The briquette, free from admixed ore, consists of ground coke and equal parts of a silicon sand and lime in sufficient quantities to serve as a binder.

H. ROYAL-DAWSON.

Production of beryllium. **Production of metals by electrolysis.** E. C. R. MARKS. From KEMET LABORATORIES Co., INC. (B.P. 287,734 and 287,762, 20.4.27).—(A) Anhydrous beryllium chloride, obtained by sublimation from a mixture of the oxide and carbon heated in chlorine, carbonyl chloride, or carbon tetrachloride, is added to a bath of molten sodium chloride and the mixture is electrolysed at 5–8 volts in an inert atmosphere, the temperature being raised slowly from 730° to 820° during electrolysis. The beryllium deposits in flakes on the anode. When electrolysis is finished the molten sodium chloride is poured out and the beryllium collected, washed free of soluble salts, dried, pressed into rods or discs, and melted under a layer of 10% of barium fluoride and 90% of barium chloride. (B) The vessel in which the electrolysis is carried out is

made of a nickel–chromium or iron–chromium alloy containing more than 5% Cr, preferably 20% Cr.

A. R. POWELL.

Manufacture [refining] of aluminium. ALUMINIUM-IND. A.-G. (B.P. 265,170, 7.1.27. Ger., 27.1.26).—Current is passed between an aluminium anode to be refined and a cathode through an electrolyte, *e.g.*, aluminium sodium chloride, of lower m.p. than the anode and cathode, whereby fusion of these electrodes is avoided.

J. S. G. THOMAS.

Production of nickel or ferro-nickel. M. STERN (B.P. 269,133, 22.11.26. Ger., 7.4.26).—Materials containing nickel or nickel oxide are melted together with iron, and the content of nickel is increased either by preferential oxidation of the iron or by the addition of further quantities of nickel.

C. A. KING.

Recovery of nickel from compound sheets of copper and nickel. E. BREUNING (B.P. 274,064, 27.6.27. Ger., 10.7.26).—Copper may be dissolved from the surface of a nickel sheet by treatment at 80° with neutral ferric sulphate solution in the presence of an oxidising agent such as air, oxygen, ozone, or a persulphate, and an oxygen carrier such as ceric sulphate. The nickel becomes passive and remains unattacked.

A. R. POWELL.

Separation of tin from alloys and mechanical mixtures. F. BISCHITZKY (B.P. 288,049, 23.5.27).—White metal alloy scrap with a high content of tin is treated in counter-current with a boiling solution of hydrochloric acid and calcium chloride until the liquid ceases to dissolve more metal. The solution deposits lead chloride on diluting and cooling, and the tin may be recovered by precipitation with lime or calcium carbonate. The residues from the acid treatment may still contain tin; they are washed free from acid, allowed to oxidise by exposure to the air, and returned to the treatment vats.

A. R. POWELL.

Treatment of impure [silver, lead, or tin] metals or alloys [for the removal of copper]. H. HARRIS (B.P. 288,004, 21.2.27).—The molten metal or alloy is treated with sulphur or a sulphur-bearing material such as hydrogen sulphide in the absence of air or in a reducing atmosphere. To cause complete separation of the copper sulphide from the purified metal, resin or other similar carbonaceous material is stirred in after the sulphur treatment.

A. R. POWELL.

Purification of metals. J. J. CONLIN, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,665,844, 10.4.28. Appl., 6.4.27).—A readily oxidisable metal may be freed from non-metallic impurities by inclosing it in a container with an upper valve, and submerging the container below the surface of a molten salt bath which does not react with the metal, but causes the impurities to separate from the metal.

A. R. POWELL.

Bearing [metal] alloy. I. R. VALENTINE, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,667,641, 24.4.28. Appl., 25.9.25).—The alloy contains 70 pts. of copper, a hardening metal, about 25 pts. of lead, and a trace of arsenic.

F. G. CROSSE.

Alloy. M. G. CORSON, Assr. to ELECTRO-METALLURGICAL Co. (U.S.P. 1,667,966, 1.5.28. Appl., 21.6.26).—

A bronze contains up to 9% Al, above 0.5% Cr in solid solution, and sufficient nickel to hold the chromium in solution.

F. G. CROSSE.

Production of a refractory material [alloy]. Alloy and material employing the same. J. G. DONALDSON and H. L. COLES, Assrs. to GUARDIAN METALS Co. (U.S.P. 1,668,306—7, 1.5.28. Appl., [A] 16.1.25, [B] 19.5.23).—(A) A copper alloy contains 0.60—0.75% Si and less than 1% C. (B) A compound plate consists of a core (an alloy containing 99.385—99.230% Cu, 0.015—0.020% C, and 0.60—0.75% Si) surrounded by other metal to which it is united, e.g., by alloying therewith.

F. G. CROSSE.

Apparatus for cleansing metals from oil and other impurities. Dr. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 273,307, 22.6.27. Ger., 25.6.26).—An apparatus for cleaning articles with organic liquids, e.g., trichloroethylene, benzene, carbon tetrachloride, consists of two closable vessels arranged one above the other. The upper vessel contains the articles in baskets which may be rocked, and a pump is used to force the cleaning liquid from the bottom chamber to the one above or in the reverse direction. Heat may be applied to the lower chamber, or a current of hot air for drying the cleaned articles, a condenser being mounted on top of the chambers.

C. A. KING.

Increasing the elasticity of metal articles. G. L. KELLEY, Assr. to BUDD WHEEL Co. (U.S.P. 1,667,476, 24.4.28. Appl., 14.9.26).—Rings produced by cold-working blank stock are heated at 93—427° for $\frac{1}{2}$ —30 min.

J. S. G. THOMAS.

Friction materials for surfacing the friction elements of brakes, clutches, etc. Soc. ANON. FRANÇ. DU FERODO (B.P. 264,471, 20.12.26. Fr., 13.1.26).—The friction surfaces are impregnated with lead or an alloy of lead by either mechanical or chemical means.

C. A. KING.

Solder bars. H. A. MEINHARDT (B.P. 282,007, 20.9.27. Ger., 10.12.26).—In a composite soft-solder bar the tubular sheath of solder has a lower m.p. than the core. The core may also contain fluxes or deoxidising substances.

C. A. KING.

Flotation processes. D., M., S. R., and S. GUGGENHEIM, J. K. MACGOWAN, and E. A. C. SMITH [GUGGENHEIM BROS.], Assees. of A. H. FISCHER (B.P. 275,561, 29.4.27. U.S., 3.8.26).—Flotation agents comprising derivatives of thiocarbonic acid, such as dioxanthogen or xanthic anhydride, or their alkyl, aryl, or aralkyl homologues, are claimed.

A. R. POWELL.

Process of welding. R. APPELEGATE, Assr. to OHIO BRASS Co. (U.S.P. 1,666,026, 10.4.28. Appl., 10.10.21).—In welding copper bands to steel rails by the arc process, calcium silicide is used as a deoxidiser.

A. R. POWELL.

Manufacture of plated goods. E. G. BEK (B.P. 287,641, 24.12.26. Addn. to B.P. 244,487; B., 1927, 633).—In the process of plating base-metal articles with a precious metal by electrodeposition followed by an annealing operation, the colour of the deposit may be modified by depositing a second layer of the same or another metal and again subjecting the article to an

annealing operation to cause diffusion between the layers to take place. To coat a gold-plated article with platinum, several repetitions of the process using an annealing temperature of 700—800° are necessary. To convert a coating of red gold into one of white gold the gold-plated article is further plated with nickel and heated for 2 min. at 700°; or into green gold by plating with a thin layer of cadmium and heating at 300—600°. A yellow gold coating is converted into one of white gold by plating with chromium and heating at 600—800°. A platinum coating may be applied to a base metal or gold article by plating first with cadmium, zinc, or silver and heating at 400°, then plating with platinum and heating at 600—700°.

A. R. POWELL.

Magnetic separation of minerals. R. RADEMACHER and E. GOEBEL (B.P. 274,889, 21.7.27. Ger., 23.7.26).—Ferrous and non-ferrous constituents of a mineral are separated by magnetic treatment of the mixture at a temperature either between 300° and 600° or above 1100°. The residue, after separation of the ferrous constituent, may be re-treated.

J. S. G. THOMAS.

Cupola furnace. O. WEICHEL and W. HOLLINDER-BÄUMER (U.S.P. 1,668,133, 1.5.28. Appl., 7.7.27. Ger., 2.6.26).—See B.P. 272,216; B., 1928, 160.

Magnetic alloy. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (U.S.P. 1,667,746, 1.5.28. Appl., 21.1.27. U.K., 8.9.26).—See B.P. 281,763; B., 1928, 128.

Magnetisable material [alloy]. E. GUMLICH (U.S.P. 1,668,115, 1.5.28. Appl., 30.11.26. Ger., 30.11.25).—See B.P. 262,153; B., 1927, 560.

[Wire]-enamelling ovens. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of C. L. HEISLER (B.P. 282,740, 12.12.27. U.S., 29.12.26).

Application of protective coatings to metal pipes etc. W. R. HUME, and HUME STEEL, LTD. (B.P. 287,656, 10.1.27).

Manufacture of electrodes, welding rods, etc. for use in arc welding and depositing metal. B. TURNER and FERRO-ARC WELDING Co., LTD. (B.P. 285,128—9, 9.11.26).

Furnace for ores (U.S.P. 1,665,546).—See XI.

XI.—ELECTROTECHNICS.

Principles governing the choice and utilisation of permanent-magnet steels. R. L. SANFORD (U.S. Bur. Stand., Sci. Paper 567, 1927, 22, 557—567).—Magnet steels with a high cobalt content have coercive force values 2—5 times as great as those of the chromium or tungsten steels generally used in the manufacture of permanent magnets, and the residual induction is only very slightly lower, but cobalt steels cannot be satisfactorily substituted for chromium or tungsten steels without changing the design of the magnet. From tests of numerous types of magnet steels it is shown that the product of the coercive force and the residual induction is the best criterion of the value of a steel for this purpose. Magnet steels can be stabilised against permanent deterioration from transient demagnetising fields by subjecting them to partial demagnetisation; this method is particularly applicable

to the treatment of cobalt steels owing to their high coercive force.

A. R. POWELL.

Influence of the cathode on the electrodeposition of chromium. H. S. LUKENS (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 7 pp.).—A solution containing 250 g. of commercial chromic acid and 3.3 g. of chromic sulphate per litre was electrolysed at 45° in a diaphragm cell for 1 hr. and the composition of the catholyte after the electrolysis was determined. Experiments were made with cathodes of electrolytic nickel, electrolytic copper, and sheet lead at various current densities. The greatest amount of trivalent chromium is produced in the catholyte at the lowest current density used (5 amp./dm.²), and at given current density the amount is least with a nickel cathode and greatest with a lead cathode, *i.e.*, it increases with increasing hydrogen overvoltage of the cathode metal. Of the three cathode metals, copper yields practical deposits of chromium over the widest range of current densities, nickel over the narrowest range. When the concentration of chromic salt in a chromium bath exceeds a low limiting value, the conductivity of the bath becomes impracticably low: such a solution can be rejuvenated by electrolysis using a very large lead anode and a small cathode surrounded by a porous diaphragm. In attempting to deposit chromium on the surface of nickel which had been electrodeposited on graphitised wax, the surface of the nickel which had been in contact with the wax was treated with an abrasive after removing the wax by application of hot water. The very narrow limits of current density required for the deposition of chromium on this surface and the behaviour of the surface towards chemical reagents indicate that it is in a passive state.

H. J. T. ELLINGHAM.

Properties of graphite used in electrotyping. J. H. WINKLER and W. BLUM (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 19 pp.).—The fineness, electrical conductivity, and carbon content of five samples of graphite were determined, and wax moulds were coated with films of graphite by application of aqueous suspensions of the various samples. The rate at which nickel deposits from a specified electrotyping solution under specified conditions on the various graphite films was determined (*cf.* Blum and Winkler, following). From such tests carried out both in the laboratory and in commercial electrotyping plants, tentative specifications for graphite for use in electrotyping are suggested. For graphite applied from aqueous suspension ("wet leads"), the material should contain at least 92% C; at least 99% should pass through 100-mesh, 95% through 200-mesh, and 50% through 325-mesh; it must readily form a suspension with water, and the film should be covered with nickel in 10 min. under the specified conditions. Slightly different specifications are given for "polishing leads."

H. J. T. ELLINGHAM.

Nickel electrotyping solutions. W. BLUM and J. H. WINKLER (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 15 pp.).—Experiments have been made on the rate of "covering" of a graphitised wax slab by nickel electrodeposited from solutions of various compositions under various conditions. Electrical

connexion to the graphitised surface was made by means of a brass strip. The physical character of the nickel deposits is recorded. The presence of an ammonium salt in the bath is found to be essential, but addition of boric acid is generally unsatisfactory in that the nickel deposit tends to crack and curl. The best nickel concentration is found to be about 0.5*N*; higher concentrations increase the tendency to crack and curl. The rate of covering increases somewhat with increasing conductivity of a bath of given nickel content; it also increases with rise of temperature, but a duller and softer deposit is then obtained. If the current density is too high, "burnt" or cracked deposits are produced, but, since the current density is changing continuously during deposition, it is usual to operate at a constant specified voltage. It is recommended that a solution which is *N* to nickel sulphate and 0.1*N* to ammonium chloride should be used, the *p_H* value being maintained at 6.0–6.4 at 25–35° in a well agitated bath. With anodes and cathodes 7.5–10 cm. apart, the voltage applied may be about 4. The use of this bath, which is more concentrated in nickel than is usual for electrotyping baths, has proved generally satisfactory in commercial operation over an extended period. Recommendations are also given for nickel deposition on lead moulds. In this case the solution may be still more concentrated and of higher *p_H* value, and can be operated at 4–5 volts at 30–45°.

H. J. T. ELLINGHAM.

Effect of temperature and other factors on the performance of storage batteries. G. W. VINAL and C. L. SNYDER (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 12 pp.).—Using small experimental batteries having either one positive plate and two negatives or one negative and two positives set up in acid of various concentrations, which, however, remained practically constant during operation, measurements of capacity were made at various rates of discharge and at various temperatures (–17° to 25°), the capacities of the positive and negative plates being separately determined. At given temperature and discharge rate, the capacity of the positive plates increases with the concentration of the acid used, but that of the negative plates decreases. Hence the best concentration of electrolyte is a compromise between the requirements of the positive and negative plates. Since under extreme conditions, as in aeroplane service, the negative plates are likely to become the limiting factor in determining the capacity, more dilute acid will then be desirable. With given acid concentration and discharge rate, a fall of temperature reduces the capacity of both plates, but the effect is greater with the negative than the positive plates. Also the temperature effect is least at higher electrolyte concentrations in the case of the positive plates, whereas the reverse is true for the negatives. In a battery where the capacity is limited by the positives, the change in capacity with temperature is inversely proportioned to the change in the viscosity of the solution. The conditions under which localised freezing may occur at lower temperatures are considered. The possible effects of these various factors on the operation of aeroplane batteries are discussed.

H. J. T. ELLINGHAM.

Dry cell service and tests. C. A. GILLINGHAM (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 6 pp.).—The processes occurring in a dry cell may be classified as (a) chemical depolarisation, which occurs only when the cell is under load, (b) physical depolarisation, including escape of hydrogen gas and diffusion processes, which occurs during and after the delivery of current by the cell, and (c) shelf reactions, including the chemical dissolution of zinc, which occur at all times. The relative importance of these three groups in determining the efficiency of the cell depends on the nature of the service in which it is employed. Tests intended to give a true indication of the suitability of a cell for various types of service are suggested.

H. J. T. ELLINGHAM.

Gelatinisation of corn starch in dry cell electrolytes. W. D. STALEY and A. J. HELFRECHT (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 7 pp.).—A mixture of 2 pts. of corn starch and 1 pt. of corn meal was added to solutions of zinc chloride or of zinc and ammonium chlorides at 10°, and after thorough mixing the time taken for the solutions to gelatinise on being kept in surroundings at 18° was determined. With 35 g. of the cereal in 80 c.c. of zinc chloride solution, the solution does not gelatinise unless the zinc chloride concentration is as high as about 35%; with increasing concentration of zinc chloride the setting time decreases to a minimum at about 42% and then increases rapidly again. Similar results are obtained with solutions containing ammonium chloride and zinc chloride, but the former salt is only about half as effective in promoting setting as compared with the weight of the latter which it replaces. By the use of two solutions, one of which contains ammonium chloride and most of the zinc chloride and the other contains ammonium chloride, a little zinc chloride, and all the cereal, mixing in suitable proportions gives a solution which will set in a convenient time for the preparation of dry cells.

H. J. T. ELLINGHAM.

Carbon dioxide recorder. GORDON and LEHMANN. —See I. **Transformer sheet iron.** VON MOOS and others. **Normalising of steel.** WOODWARD. **Tungsten steel.** SWAN. **Annealing non-ferrous metals.** KEENEY. **Cable sheathing.** BECKINSALE and WATERHOUSE. **Speiss and metals of platinum group.** RUSDEN and HENDERSON.—See X.

PATENTS.

Electric furnace. H. WIGGIN & Co., LTD., and A. G. LOBLEY (B.P. 285,147, 11.11.26).—Portions of the shanks of resistance-suspender or connectors are, after insertion through the furnace bricks, bent over to form keys which are engaged with hook-locating grooves or seatings formed either in the brick or in bonded or non-rotatable sleeves or insulators applied to the brick.

J. S. G. THOMAS.

Electric furnace. E. C. SASNETT, Assr. to C. B. FOLEY, INC. (U.S.P. 1,660,209, 21.2.28. Appl., 24.9.19. Renewed 7.7.27).—A structure lined with refractory material is adapted to hold molten metal in the shape of a superposed pool with a subjoined loop, the cross-sectional area of which is a minimum at one point at which it joins the pool and increases gradually and con-

tinuously to a maximum at the other. Electrical induction means are threaded through a transverse opening within the loop.

J. S. G. THOMAS.

Electric furnace. A. J. ASCH (U.S.P. 1,661,026, 28.2.28. Appl., 4.2.27).—A heating element composed of platinum foil is supported within the furnace, and a source of current is connected to portions thereof extending outside the furnace wall.

J. S. G. THOMAS.

Electric furnace for treating ores, shales, etc. F. E. HATCH (U.S.P. 1,665,546, 10.4.28. Appl., 4.9.26).—An electrically heated, horizontal, rotatable drum is lined with refractory material. The heads of the drum are closed and have inlet and exit for material. Pipes pass through the drum to a collector at one end, and means are provided for causing a current of air to pass through the pipes and collector.

T. A. SMITH.

Ironless induction furnaces or heating apparatus. SIEMENS & HALSKE A.-G. (B.P. 274,888, 21.7.27, and Addn. B.P. 275,249, 28.7.27. Ger., [A] 24.7.26, [B] 29.7.26).—(A) Heat produced by induction in the charge is generated by a heating coil dipped or inserted into the charge in the furnace interior so that a large part of the external electromagnetic lines of force produced by the heating coil pass through the charge. (B) In addition to the heating coil dipping into the charge, another coil is arranged round the outside wall of the furnace.

J. S. G. THOMAS.

Recovery of reaction products from gases treated with electric arcs. I. G. FARBERIND, A.-G. (B.P. 263,859 and Addn. B.P. 288,056, 30.12.26. Ger., 30.12.25).—(A) Small outlet tubes or pipes are arranged in combination with gas conduits in order to separate gas rich in reaction products from gas weak in reaction products. (B) The process is applied for producing pure hydrogen from mixtures containing hydrogen and methane which are treated in electric arc furnaces whereby the methane is largely converted into acetylene. The reaction products are separated into a portion containing practically no unconverted methane and a portion containing very little acetylene. The process is also applied to the separation of oxides of nitrogen produced by the fixation of atmospheric nitrogen.

J. S. G. THOMAS.

Net-cathode for electrolytic cells. A. ERGANG (B.P. 287,760, 11.7.27).—The wire gauze net-cathodes used, e.g., in electrolysing alkali chlorides, are replaced by cathodes formed from sheet metal by perforating with rectangular holes by means of a punch so constructed that the upper surfaces of the bars between the holes are rounded.

[Electrolyte for] voltaic cells. FRIGAMIN G.M.B.H. (B.P. 283,559, 5.7.27. Ger., 14.1.27).—Hydrochlorides of the organic amines, more especially methylamine hydrochloride, to which may be added one or more weak organic acids, e.g., cinnamic acid, sulphonic acids, are employed as electrolytes in cells of the Leclanché type.

J. S. G. THOMAS.

Production of a storage-battery plate. C. W. JENNER (U.S.P. 1,665,962, 10.4.28. Appl., 2.6.27).—Oxidised, finely-ground electrolytic lead is mixed with dilute sulphuric acid and moulded into a plate.

J. S. G. THOMAS.

Glow cathode and electron tube containing same. E. FRIEDERICH, Assr. to RADIO CORP. OF AMERICA (U.S.P. 1,667,471, 24.4.28. Appl., 28.11.25. Ger., 1.12.24).—A highly refractory filamentary core coated with a mixture of carbon and a metal of high electron-emitting capacity is heated so that a carbide of the metal is formed. J. S. G. THOMAS.

Magnetic separation. F. M. SIMONDS, Assr. to A. F. HYDE (U.S.P. 1,660,362, 28.2.28. Appl., 3.10.25).—A mixture comprising magnetic and non-magnetic particles moves across a magnetic field, and the aggregates formed are subjected to disintegrating blows as they move towards the source of magnetic attraction. J. S. G. THOMAS.

Treatment of magnetisable materials. A. F. BANDUR, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,666,191, 17.4.28. Appl., 12.12.24).—Magnetisable material is locally flash-heated to a high temperature and cooled, the rate of cooling being controlled so that there is a rapid loss of heat from a temperature not below 500°. J. S. G. THOMAS.

Treatment of wax surfaces [for electrotype] with graphite. W. McLAUGHLIN and H. KNOWLES (B.P. 288,709, 11.1.27).—Wax surfaces are treated with a colloidal suspension of graphite, preferably a suspension in water containing a protective colloid, *e.g.*, tannic acid, and a deflocculating agent, *e.g.*, sodium silicate, glycerin, or eucalyptus oil. Thus, "Aquadag" may be employed. J. S. G. THOMAS.

Gas-testing apparatus. C. ENGELHARD, INC., Assees. of R. H. KRUEGER (B.P. 288,908, 17.10.27. U.S., 27.8.27).—In gas-analysis apparatus of the catharometer type, controllable means, *e.g.*, tubular insulating thimbles, are provided in order to regulate the heat dissipated in the chambers containing gases and the resistances forming arms of the Wheatstone bridge. J. S. G. THOMAS.

Lead accumulator. R. OPPENHEIM, Assr. to SOC. ANON. LE CARBONE (U.S.P. 1,667,435, 24.4.28. Appl., 18.4.25. Fr., 14.6.24).—See B.P. 235,530; B., 1926, 591.

Use of gas-filled photo-electric cells. GEN. ELECTRIC Co., and N. R. CAMPBELL (B.P. 288,747, 24.1.27, and 288,783, 25.2.27).

Electric mercury-vapour apparatus [rectifier] etc. GEN. ELECTRIC Co., LTD., and E. WEINTRAUB, Assees. of SOC. ALSACIENNE DE CONSTRUCTIONS MÉCANIQUES (B.P. 272,958, 20.6.27. Fr., 19.6.26).

Beryllium (B.P. 287,734 and 287,762). **Refining of aluminium** (B.P. 265,170). **Plated goods** (B.P. 287,641).—See X.

XII.—FATS; OILS; WAXES.

Bleaching of [linseed] oil. K. WÜRTH (Farben-Ztg., 1928, 33, 1852—1853).—Linseed oil was treated with ozonised air, a large, moving surface of oil being exposed during the process. The oil was rapidly bleached and its consistency unchanged, but, on gentle heating, the oil assumed a brownish colour and a strongly smelling gas was evolved. Similar results were obtained by treating the oil with air while exposed to ultra-

violet light from a mercury lamp. The presence of moisture in the air or in the oil may influence the processes involved. S. COFFEY.

Standardisation of cod-liver oil. W. H. DICKHART (Oil and Fat Ind., 1928, 5, 82—84).—A rapid sorting test, to distinguish good oils from those contaminated with oils other than those from the livers of fish related to the cod, consists in adding 10 mg. of uranium nitrate to 3 c.c. of oil placed in a 6-in. glass tube of inside diam. 20 mm. The tube is placed upright in a 400 c.c. beaker containing 80 c.c. of water which is heated just at the b.p. for 20 min.; afterwards the oil is allowed to cool and the colour measured in a Wesson tintometer using standard Lovibond glasses. Experiments and analyses proved that fish oil changed to give a definite colour reading with uranium nitrate. Comparison of data on colour readings for a number of commercial cod-liver oils showed but one passing the U.S.P. test for unsaponifiable matter, and that oil gave characteristics similar to whale oil, and yet all the oils were cod-liver oils. It is suggested that for medicinal cod-liver oil a maximum of 1 yellow, 0.1 red before heating with uranium nitrate and 35 yellow, 3.8 red after heating be allowed on a basis of a 10 mm. × 20 mm. column of oil; that for poultry oil 1.5 yellow, 0.2 red in the cold and 35 yellow, 5.0 red after heating be the established standard in the uranium nitrate test conducted as described. H. M. LANGTON.

Analysis and technology of evening primrose oil. A. EIBNER and E. SCHILD (Chem. Umschau, 1927, 34, 339—342; cf. Heiduschka and Lüft, B., 1919, 426 A).—The oil has d_{4}^{15} 0.9312; viscosity (Engler) at 24° 8.14, n_D^{20} 1.47154, acid value 4.51, saponif. value 187.39, iodine value (Hanus) 147.89, glycerin content 10.01%, unsaponif. matter 1.60%, total fatty acids 95.52% (iodine value 156.65). Separation of the fatty acids (Twitchell) gave 5.37% of saturated acids and 94.63% of unsaturated. Analysis of that part of the unsaturated brominated acids soluble in light petroleum shows the existence of an isomeric γ -linolenic acid. The results obtained are (%): γ -linolenic acid 2.69, isomeric γ -linolenic acid 6.00, α -linolenic acid 29.55, β -linolenic acid 22.23, oleic acid 23.56, saturated acids 5.13, unsaponifiable matter 1.60, and glyceryl radical 4.11. This oil does not attain the drying properties of the pine seed oils, which, according to Reitter (B., 1926, 593), contain 6.6% of α -linolenic acid and 17.2% of β -linolenic acid. Since γ -linolenic acid as a symmetrical molecule has not such strong drying qualities as the α -linolenic acid of perilla and linseed oil, quantitative analysis of an oil throws light on its drying qualities, in which respect it is shown to be midway between linseed and poppyseed oil. H. M. LANGTON.

Chemical composition of ergot oil. W. F. BAUGHMAN and G. S. JAMIESON (Oil and Fat Ind., 1928, 5, 85—89; cf. Rosenheim and Webster, A., 1927, 1224; Dieterle, Diester, and Thimann, B., 1927, 799; Matthes and Schütz, B., 1927, 891).—The ergot oil examined was extracted by light petroleum from a composite sample of Russian, Spanish, and Austrian ergot containing 5.50% of moisture and 32.75% of oil.

The oil, dark red by transmitted light with a dark brownish yellow fluorescence by reflected light, had d_{25}^{25} 0.9222, n_D^{25} 1.4691, acid value 3.02, iodine value (Hanus) 73.8, saponif. value 196.9, unsaponifiable matter 1.18%, acetyl value 7.3, R.-M. value 0.3, Polenske value 0.4, saturated acids 27.2% (iodine value 2.6), unsaturated acids + unsaponifiable matter 68.6%, iodine value of unsaturated acids 101.2, saturated acids (corr.) 26.5%, unsaturated acids (corr.) 68.1%. The composition of the unsaturated acids was determined from their saponif. and iodine values, that of the saturated acids being determined by fractional distillation of their methyl esters. The composition of ergot oil is summarised thus: glycerides of oleic acid 62.5%, of linoleic acid 8.7%, of myristic acid 0.3%, of palmitic acid 21.5%, of stearic acid 5.3%, of arachidic acid 0.7%, unsaponifiable matter 1.2%. No confirmation was obtained of the presence of daturic acid or of hydroxylated acids previously reported in the oil.

H. M. LANGTON.

Determination of sodium sulphate in sulphonated oils. W. HERBIG (Chem. Umschau, 1927, 34, 330—331).—The oil (10 g.) is shaken with 200 c.c. of absolute alcohol in an Erlenmeyer flask which is then lightly closed. On keeping, the sodium sulphate separates very quickly as a yellowish-brown precipitate which, after decanting the alcoholic solution, is washed on to a filter with absolute alcohol. The precipitate is then dissolved in hot water and converted into barium sulphate in the usual way. Two different qualities of oil gave sodium sulphate (as SO_3) (a) 1.97 and 2.05%, (b) 0.68 and 0.75%.

H. M. LANGTON.

Deflocculation and detergency. R. M. CHAPIN (Oil and Fat Ind., 1928, 5, 95—106).—Following on his earlier work (cf. B., 1927, 117; 1928, 4) on the deflocculation of carbon black, the author has experimented on fabrics soiled by the same substance with the object of determining whether deflocculation and detergency are correlative phenomena. A laboratory washing machine is described, also a Kober-Klett colorimeter which had been converted into a reflectometer for the evaluation, by numerical expression, of soil remaining on a fabric. Detergent experiments were made on new cotton fabric (previously cleansed by boiling with soap) soiled in simple aqueous suspensions of non-oily carbon black. The most powerful detergent appeared to be an alkaline solution of sodium stearate or palmitate at 70° or above. In this effect alkali deflocculation and detergency are parallel, but other possible correlative factors were only vaguely defined. The method is not sufficiently selective of the superior amongst competing commercial soaps. Comparative tests were made on the power of soap solutions to prevent adsorption of suspended carbon black by clean fabric. Acid soaps were the most effective, probably owing to masking of adsorptive affinities by an oily film of fatty acid. Power to prevent adsorption appears not to be correlative with detergent power in the special case of the prevention of adsorption of suspended carbon by fabric. This result is based on tests using soap solutions.

H. M. LANGTON.

Rancidity determinations and a possible source of error in the Kreis test. W. C. POWICK (Oil and Fat Ind., 1928, 5, 107—108).—In the examination of fats and oils for rancidity by the Kreis test with hydrochloric acid and phloroglucinol, misleading results may be obtained through the presence of nitrosyl chloride in the acid. The acid so contaminated gives a "blank" test with phloroglucinol, and under such conditions rancid fats will generally appear to be sweet, whilst sweet fats may occasionally appear to be rancid unless the positive reaction be controlled spectroscopically. In the absence of spectroscopic control, positive tests obtained from cottonseed oils are not sufficient evidence of rancidity (cf. B., 1923, 376 A).

H. M. LANGTON.

Precision of iodine-bromine value determinations. L. W. WINKLER (Arch. Pharm., 1928, 266, 189—193).—The latest official (German) method of determining iodine values, in which iodine is used only as an indicator, gives very trustworthy results. The error in repeated determinations is less than 0.5%. The determination is rapidly carried out, but the time allowed for interaction should be adjusted according as the iodine value of the oil under examination is low or high, i.e., for iodine values below 40, 10 min. suffices; for those between 40 and 140, $\frac{1}{2}$ —1 hr.; and so on (tables are given). Cod-liver and linseed oils are exceptional, and special tables are given for these. The method has the further advantage of using cheap reagents.

W. A. SILVESTER.

Behaviour of beeswax towards trichloroethylene at ordinary temperatures. G. BUCHNER (Chem.-Ztg., 1928, 52, 319).—Treatment in the cold causes the removal of a large portion of the hydrocarbons present in the beeswax. The soluble portion has m.p. 54.5°, acid value 3.45, ester value 24.36, and saponif. value 27.8, whilst the corresponding values for the insoluble fraction are 67.5°, 15.6, 54.0, and 69.6. The ratio of cerotic acid to ester in the original wax was 1:6.5, that in the soluble portion 1:12, and in the insoluble portion 1:6.3.

H. INGLESON.

Examination and evaluation of beeswax. E. ELSER (Schweiz. Chem.-Ztg., 1928, 27—32, 40—45).—A precise method of determining m.p. of waxes, using a 1.5 mm. capillary tube in a bath, the temperature of which is raised 4° per min. from an initial temperature not above 10—15°, is described. Tables are given showing the variation of m.p. of three fractions of beeswax when mixed with varying amounts of carnauba wax, ceresin, Japan wax, spermaceti, and paraffin waxes (m.p. 58° and 48°). Each wax is considered in some detail and its characteristic effect on the m.p. of beeswax demonstrated in a series of curves. The author concludes that modern chemical and physico-chemical research has shown that the thermal method of analysis of beeswax is simple and exact, and permits the detection not only of the nature but the percentage of adulteration.

E. HOLMES.

PATENTS.

Purification of olive oil and other edible oils. E. FORAY (Addn. No. 31,181, 17.11.25, to F.P. 605,389; B., 1926, 795).—The oil, after neutralisation and addition

of salt, is washed three times with half its weight of boiling water to remove traces of dissolved soaps, treated at 60° with 2% of regenerated fuller's earth (or with 1.5—3.0% of fresh earth for dark-coloured oil), passed hot through a filter press, and deodorised with 0.05% of manganese dioxide. L. A. COLES.

Bleaching of fatty substances of vegetable and animal origin. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 286,794, 11.12.26).—A combination of the oxidising and reducing processes for bleaching is claimed to give improvements in the colour and odour of fatty materials. The substances may be subjected to a reducing bleach immediately preceded or followed by treatment with one or more inorganic, non-gaseous, oxidising agents, or it may be treated with an absorptive agent simultaneously with the oxidising agent. Fatty substances so treated may be saponified and then bleached with hypochlorite to give light-coloured soaps. Where there is a tendency to form emulsions, from which the manganese dioxide or chromic hydroxide can be removed only with difficulty, it is an advantage to add an equivalent of mineral acid at the outset. E. HOLMES.

Sulphonation of fatty acids and their esters. H. T. BÖHME A.-G. (B.P. 261,385, 10.11.26. Ger., 11.11.25).—Difficulties arising from the formation of water in the sulphonation of fatty acids and their esters (*e.g.*, castor oil) by sulphuric acid are obviated by carrying out the reaction in the presence of anhydrous organic acids or their anhydrides or chlorides, especially acetic acid. B. FULLMAN.

Production of strongly frothing soaps. CHEM. FABR. STOCKHAUSEN & Co. (G.P. 445,848, 9.6.25).—A mixture of about equal parts of turpentine with perchloroethylene or other chlorinated hydrocarbons is added to the usual ingredients of soap. L. A. COLES.

Production of stable moistening and degreasing agents. G. ZIMMERLI CHEM.-TECHN. FABR. (SWISS P. 119,114, 23.3.26. Addn. to Swiss P. 111,767).—A mixture of hydrogenated naphthalene with sulphonated fat having a sulphonic acid content of over 40% of the total fats is neutralised and emulsified with water, yielding a product not affected by hard water and possessing good emulsifying, degreasing, and purifying properties. L. A. COLES.

Purification of fat solvents after use. R. A. A. JEANNIN (F.P. 622,238, 28.9.26).—Fat solvents, *e.g.*, benzol, benzine, and trichloroethylene, are treated successively with sand acidified with sulphuric acid and sand made alkaline with sodium carbonate solution, the solvent being decanted off from the residue after each treatment. L. A. COLES.

Alkali fusion [saponification of glycerides]. V. R. KOKATNUR (U.S.P. 1,667,480, 24.4.28. Appl., 22.1.26).—See B.P. 251,290; B., 1927, 755.

Coagulation or peptisation (B.P. 287,943).—See III. Products from cashew nut-shell oil (B.P. 262,134).—See XIV.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Analyses of paints with plumbago base. R. JACQUES (Ann. Chim. analyt., 1928, [ii], 10, 98—100).—A 25 g. sample was repeatedly extracted with ether and

filtered through a tared Gooch crucible. The residue was then washed with 95° alcohol to extract resins, the precipitate was dried at 105°, and the filtrate evaporated at 130—140°. If the solid colouring matter and the oil so determined show a deficiency from 100%, the difference is probably light spirit. This can be determined by distilling a sample with four times the weight of water. The solid base was tested for loss on ignition and another portion extracted with hydrochloric acid and the residue weighed in a Gooch crucible. If carbonates are present they must be separately determined; otherwise the loss on ignition gives the proportion of graphite present. The analysis of the insoluble residue for silica and metallic bases was completed in the usual way. C. IRWIN.

Mechanism of the drying of red lead and white lead pigments. W. VAUBEL (Z. angew. Chem., 1928, 41, 181—183).—In the drying of pigments prepared from red lead and white lead and linseed oil, although the oxidation of the linseed oil to linoxyn is the main process, subsidiary reactions occur as follows:—(1) neutralisation of the free acids present in the oil by the lead pigment, (2) hydrolysis of the linseed oil by the lead compound, affording glycerol and lead linoleate, (3) reaction between the lead compound and glycerol, (4) neutralisation by the lead compound of the mono- and diglycerides present or formed in the drying process, (5) neutralisation of the acetic acid or formic acid formed by oxidation in the drying process. Such hydrolysis of the linseed oil may be a source of streakiness in the pigment, owing to the hygroscopic character of the glycerol liberated, and the presence of glycerol so formed constitutes a danger to the effectiveness of the "anti-rust" pigments containing lead, white lead and red lead reacting only very slowly with glycerol. Litharge reacts rapidly with glycerol, affording a solid compound, $C_3H_5(O \cdot PbOH)_3$, which on heating at 120—130° to constant weight is converted into $Pb[O \cdot C_3H_5(OPb)_2O]_2$, which can be used in the determination of glycerol. Lead hydroxide reacts very slowly, the reaction being accelerated by heat. Lead peroxide does not react cold, but reacts rapidly when heated; lead sulphate, barium sulphate, and lithopone are without action on glycerol. Zinc oxide reacts more slowly than red lead, but faster than white lead. In addition to their advantages in covering power, lead pigments thus possess advantages in their reactivity towards decomposition products formed in the drying process. R. BRIGHTMAN.

Detection of sulphur and sulphurous acid in mineral colours. A. NOLL (Farben-Ztg., 1928, 33, 1849—1850).—The colour is ignited with sulphur-free iron powder and the melt dropped into dilute hydrochloric acid, when the production of hydrogen sulphide indicates sulphur. The test gives positive results, no matter how the sulphur is combined. The presence of sulphite is ascertained by digesting the colour with hot distilled water and filtering. Sulphurous acid is detected in the filtrate either by reduction with zinc and dilute acid to hydrogen sulphide or by reduction with zinc dust alone to hyposulphite. The latter is detected by its action on indanthrene-yellow G, methylene-blue, or sodium anthraquinone- β -sulphonate. S. COFFEY.

PATENTS.

Manufacture of a dispersing powder. E. R. ALLEN and W. E. KAUFMANN, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,665,945, 10.4.28. Appl., 3.7.25).—An inorganic pigment not readily miscible with water is agitated with water and a foam-producing dispersing agent, and the resulting foam is dried and ground. The product readily disperses in water to a smooth pulp. F. G. CLARKE.

Manufacture of condensation products of urea and formaldehyde. I. G. FARBERIND. A.-G. (B.P. 288,346, 1.10.26. Addn. to B.P. 258,289; B., 1928, 376).—Carbamide and formaldehyde are condensed in a solution maintained by buffer compounds at p_H 4–6, and the product is evaporated or dehydrated at p_H 6–7. Clear products are thus obtained with comparatively small quantities of formaldehyde, and when the molar ratio of formaldehyde : carbamide is less than 2 : 1 (1.5–1.7 : 1) the evaporation or dehydration may be dispensed with partly or entirely, the condensation product separating on cooling (preferably while adding salts) as a liquid colloid. B. FULLMAN.

Condensation products. "SILUR" TECHN. U. CHEM. PROD. GES.M.B.H. and I. THORN (B.P. 287,727, 9.4.27).—Plastic condensation products are obtained by mixing carbamide or thiocarbamide with formaldehyde or compounds yielding it, and then adding starch (or substances containing it, especially flour) in a quantity which is a multiple of the weight of carbamide. Resins, fillers, etc. may be added. B. FULLMAN.

Furfuraldehyde resins. Furfuryl alcohol resins. Furan derivatives. Resinous substance. J. P. TRICKEY and C. S. MINER, Assrs. to QUAKER OATS CO. (U.S.P. 1,665,233–7, 10.4.28. Appl., [A, B] 26.5.24, [C] 31.5.24, [D] 5.7.24, [E] 23.8.24. Renewed [A, B, D, E] 27.2.28).—Furfuraldehyde is catalytically treated with (a) a metallic salt, (b) a metal, to yield a resin. (c) Treatment of furfuryl alcohol alone yields an infusible and insoluble resin. (d) Furfuraldehyde, on treatment with alkali, yields furfuryl alcohol and a salt of pyromucic acid. On acidification and heating, the pyromucic acid is set free and the alcohol resinified. (e) Mixtures of furan derivatives individually capable of resinification are converted into compound resins (e.g., an alcohol and an aldehyde). B. FULLMAN.

Water-resistant polyhydric alcohol-carboxylic acid resins. C. R. DOWNS and L. WEISBERG, Assrs. to BARRETT CO. (U.S.P. 1,667,197–1,667,200, 24.4.28. Appl., [A] 22.12.20, [B–D] 7.9.21).—See B.P. 173,225; B., 1923, 667 A.

Accelerator for hardening phenol-urea products. H. V. POTTER and J. W. CRUMP, Assrs. to DAMARD LACQUER CO., LTD. (U.S.P. 1,667,675, 24.4.28. Appl., 11.9.26. U.K., 23.10.25).—See B.P. 264,601; B., 1927, 228.

[Lacquer-]enamelling furnaces. INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 283,591, 14.1.28. Ger., 15.1.27).

Coagulation and peptisation (B.P. 287,943).—See III. **Colour lakes** (B.P. 270,750).—See IV. **Pigments** (B.P. 270,658).—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Rotatory powers and melting points of the resinous constituents of gutta-percha, balata, and allied gums. S. MINATOYA and H. KANEKO (Res. Electrotech. Lab., Japan, 1928, No. 223, 18 pp.).—The resinous constituents were separated into three divisions, fluavile, spherical albane, and needle albane. The m.p. of the fluaviles of gutta-percha lie between 37° and 90°, whilst those from balata are liquid. As a rule the specific rotatory powers of the fluaviles of gutta-percha are higher than those of the corresponding constituents from balata. The albanes are present only in small quantities. S. COFFEY.

Elastic behaviour of india-rubber. G. B. DEODHAR and D. S. KOTHARI (Indian J. Physics, 1928, 2, 305–318; cf. Phil. Mag., 1923, [vi], 45, 471).—A dynamical method is described for determining the variation of the modulus of rigidity of rubber with stress. Tests on three samples showed that the rigidity increases linearly with stress up to breaking-point. A qualitative study of the effects of heat shows that a stretched band, when heated to about 100°, contracts, but if the heating is stopped the band extends until its length exceeds that before heating, and this extension is permanent. It is possible that, just after heating, the rigidity falls considerably, but after some time it regains almost its initial value at that load. The logarithmic decrement, unlike that of a metal, decreases with the load. The behaviour of metals is similar, the difference being one of magnitude only. In variation of "torsional stiffness" with load, the behaviour of rubber seems to resemble that of annealed wire. M. S. BURR.

PATENTS.

Porous soft and hard vulcanised rubber goods. H. ZIEGNER (B.P. 283,566, 21.11.27. Ger., 14.1.27).—An absorbent filler such as sawdust saturated with a swelling agent, e.g., benzene, is introduced rapidly into a mixture of rubber with the usual compounding ingredients. The absorbed solvent causes the rubber on the surface of the particles to swell, and during subsequent drying the swollen rubber shrinks, producing capillaries. The porosity of the vulcanised product may be increased by filling the capillaries before or during vulcanisation with a non-swelling substance such as water. D. F. TWISS.

Production of rubber goods directly from latex. DUNLOP RUBBER CO., LTD., G. W. TROBRIDGE, and E. A. MURPHY (B.P. 287,946, 20.10.26).—Articles are manufactured by immersing in an aqueous dispersion of organic material, such as rubber, a hollow porous mould which has previously been dipped in a liquid, e.g., a solution of calcium chloride or other coagulant. Suction may be applied to the interior of the mould during the immersion in the dispersion to expedite deposition of the material and/or after subsequent removal to hasten drying. Completion of drying and vulcanisation are then effected by known processes. D. F. TWISS.

Production of articles from rubber and other thermoplastic substances. A. T. GUSTAFSON (U.S.P. 1,665,355, 10.4.28. Appl., 4.1.26. Swed., 12.2.25).—In the production of moulded articles from rubber and other materials which become plastic on heating, a thin

layer of cellulose derivatives is placed between the mould and the material. L. A. COLES.

Vulcanisation of rubber substances. GRASSELLI CHEM. Co., Assees. of W. B. BURNETT and I. WILLIAMS (B.P. 265,930, 29.12.26. U.S., 12.2.26).—Vulcanisation is accelerated by a condensation product of a primary amine, *e.g.*, *n*-butylamine, aniline, or *o*-tolylidiguamide, with more than a bimolecular proportion of an $\alpha\beta$ -saturated aldehyde, *e.g.*, propaldehyde, *n*-butaldehyde, or heptaldehyde, prepared in the presence of a weak organic acid, *e.g.*, acetic, stearic, or salicylic acid, as condensing agent. D. F. TWISS.

Treatment of pulverulent [rubber] fillers and pigments having a carbon base, particularly lamp blacks, carbon blacks, etc. COMP. LORRAINE DE CHARBONS, LAMPES, & APPAREILLAGES ELECTRIQUES (B.P. 270,658, 28.3.27. Fr., 10.5.26).—The particles of lamp black, carbon black, and similar carbonaceous powders are coated with a very thin covering of rubber, *e.g.*, by mixing with 1% of dissolved rubber and subsequently removing the solvent. Black so treated shows no objectionable dustiness, is compact for storage and carriage, and can be incorporated in rubber without difficulty. D. F. TWISS.

Manufacture of products [artificial rubber] from cashew nut-shell oil. M. T. HARVEY (B.P. 262,134, 27.11.26. U.S., 28.11.25).—Artificial rubber compounds may be prepared by heating cashew nut-shell oil and glycerin in varying proportions and for different periods to a minimum initial temperature of 240°. The thickened material, having the general appearance of rubber, may be vulcanised to give an elastic product which is soluble in the usual commercial rubber solvents. E. HOLMES.

Coagulation and peptisation (B.P. 287,943).—Sec III.

XV.—LEATHER; GLUE.

Practical glass extractor [for tanning materials]. R. W. FREY and H. C. REED (J. Amer. Leather Chem. Assoc., 1928, 23, 124—128).—The extractor consists of a glass tube 2½ in. diam. and 8¾ in. long, closed at its lower end. A central vapour tube 10½ in. long projects through the bottom, so that only 3½ in. remains outside it. This tube is closed at the top, and has two vapour outlets near the top and two ears near the bottom of the large tube. A further side tube is fitted into the large tube as near the bottom as possible. In use, a pad of cotton wool covers the open ends of the ears, upon which the tanning material rests. A filter-paper thimble placed over the vapour tube acts as a spreader for the condensed vapours. For extraction without refluxing, the extract is led off through the side tube. By sealing the side tube the extract collects in the large tube and flows through the ears into the boiling flask to which it is attached. A condenser on the top of the large tube completes the apparatus. D. WOODROFFE.

PATENTS.

Preparation of mineral acid-free synthetic tanning agents. J. R. GEIGY A.-G. (B.P. 276,014, 12.8.27. Ger., 16.8.26).—Synthetic tanning agents obtained from aromatic hydroxysulphonic acids and formaldehyde are

neutralised with sodium hydroxide, dried, and 100 pts. of the dry product are mixed with a fluosilicate, thereby liberating the tanning acid. D. WOODROFFE.

Production of aromatic sulphonic acids for use as tanning agents. I. G. FARBENIND. A.-G., Assees. of A. STEINDORFF, K. DAIMLER, and G. BALLE (G.P. 445,569, 6.5.23).—The products are obtained by the sulphonation of diarylmethylene ethers prepared from mononuclear phenols. Suitable material for sulphonation includes methylene diphenyl ether and the product obtained by the condensation of methylene chloride with low-boiling low-temperature tar phenols. L. A. COLES.

Manufacture of leather dressings. BRIT. DYE-STUFFS CORP., LTD., T. H. FAIRBROTHER, and A. RENSHAW (B.P. 287,222, 14.12.26).—A small amount (0.05%) of the alkali salt of a polyhalogenated phenol, particularly 2 : 4 : 6-trichlorophenol, is added to leather dressings to prevent mildew or other fungoid growths. D. WOODROFFE.

Tanning of hides and skins. E. G. STIASNY and B. JALOWZER (B.P. 287,221, 14.12.26).—Complex ferri-sulphite compounds or substances forming the same, with or without the joint use of other substances used in the tanning industry, are utilised. These complex compounds may contain an organic acid radical, and/or organic compounds, especially carbohydrates. An example is given of a product prepared by mixing 1 mol. of ferric chloride with 1—5 mols. of sodium sulphite. D. WOODROFFE.

Production of a waterproofing bottom-filling composition for boots, shoes, etc. H. BROOMFIELD, and SHOE INVENTIONS, LTD. (B.P. 287,206, 7.12.26).

Coloured leather. A. C. LAWRENCE LEATHER Co., Assees. of C. P. KELLEY and E. W. WHITE (B.P. 262,780, 7.12.26. U.S., 8.12.25).

Fast dyeings (B.P. 287,651).—See VI. Diffusion process (B.P. 274,131).—Sec XVII.

XVI.—AGRICULTURE.

Determination of the need of soils for phosphoric acid relative to the soluble silica content. A. NEMEC (Compt. rend., 1928, 186, 1060—1062).—The percentage increase in crops of various roots, grain, tubers, and grass has been correlated with the mean content in soluble silica [determined colorimetrically either by the method of Bell and Doisy (A., 1920, ii, 769) or of Atkins (B., 1924, 483)] of the cultivated and sub-soil. On the basis of 200 results it is found that when the mean silica content is greater than a certain minimum value, the addition of phosphorus as superphosphate has scarcely any effect on the crop yield. The following minimum values are found: sugar beet 12, wheat 11, barley and potato 10, rye 9, oats 7, and grass 6 mg./kg. of soil. J. W. BAKER.

Rapid determination of the phosphoric ion in soils and manures by coeruleomolybdimetry. G. DENIGÈS (Compt. rend., 1928, 186, 1052—1054).—If the phosphoric ion is water-soluble the determination is made on the diluted filtered extract by the author's method (A., 1928, 263), any colour being balanced by means of a similar boiling, acidified extract placed in

front of the standard at the moment of matching. Otherwise 50 c.c. of a 2% solution of nitric acid may be used to extract 5 g. of soil, and the extract diluted 50 times. If more concentrated acid is required 1 c.c. of extract must be evaporated, and the residue calcined and extracted with boiling acidulated water. For soils containing 1–2% Fe 12 drops of reagent are used, but greater amounts (up to 50%) must be reduced by the preparation of the reagent *in situ* in the presence of 0.1 g. of copper turnings which serve to re-reduce the reagent after it has itself reduced the iron. Iron should be added to the standard. J. GRANT.

Sulphur cycle in soil. A. RIPPEL (J. Landw., 1928, 76, 1–10).—The sulphur cycle in soil resembles the nitrogen cycle. Mineralisation of humus sulphur takes place slowly. The addition of soluble carbohydrate to a compost soil causes the water-soluble sulphate to disappear. This change is occasioned by the growth of fungus mycelia, and can be demonstrated in pure culture. It is claimed that *Aspergillus* is capable of oxidising elementary sulphur, and that the sulphuric acid produced by micro-organisms substantially influences soil acidity and rock decomposition. H. J. G. HINES.

Measurement of suction power in the seedling stage. K. MEYER (J. Landw., 1928, 76, 11–24).—A preliminary communication describing modifications of Buchinger's apparatus and procedure. Seedlings are grown on glass lattices above sugar solutions in a shallow zinc tank. The changes in sugar concentration are determined polarimetrically. H. J. G. HINES.

Relation between mechanical composition and hygroscopic coefficient of a soil. F. GIESECKE (J. Landw., 1928, 76, 33–40).—No relation could be found between the hygroscopic coefficient and the clay content or the ratio of clay to fine silt.

H. J. G. HINES.

Extraction of soils by the hydrochloric acid method. E. BLANCK and A. RIESER (J. Landw., 1928, 76, 25–31).—Three modifications of the method proposed by the International Commission were compared with the Commission's method and substantial differences observed in the subsequent analyses. The method of preparation of the soil for analysis also affected the proportion dissolved by hydrochloric acid.

H. J. G. HINES.

Determination of chlorophyll by Willstätter's method. K. MAIWALD (J. Landw., 1928, 76, 63–69). T. STECHE (*ibid.*, 71–73; cf. B., 1928, 136).—(A) An adverse criticism of Steche's modification of Willstätter's method and a description of the procedure recommended by the author. (B) A reply. H. J. G. HINES.

Influence of stimulants on the sprouting of potatoes. W. VON VELSEN (J. Landw., 1928, 76, 41–62).—Chemical and physical methods of stimulating the sprouting of three varieties of potatoes were tested. The physical methods used were ineffective, but a number of chemical agents hastened the sprouting. Treatment with hydrocyanic acid, thiourea, ethylene dichloride, and potassium thiocyanate, or injection of diastase gave marked effects. The number and total weight of

sprouts were increased also by these reagents. On following the development in spring and summer, however, no differences could be observed between treated and untreated tubers. H. J. G. HINES.

Citric acid-soluble phosphate in basic slag. A. SÜLLWALD (Arch. Eisenhüttenw., 1927–8, 1, 565–570; Stahl u. Eisen, 1928, 48, 547–548).—Basic slags containing sufficient calcium silicate to form the double compound with calcium tetraphosphate are unaffected by the rate of cooling under ordinary conditions, but rapid quenching or granulation in water causes a decrease in the citric acid-soluble phosphate content. When the slag is cooled in large blocks the total phosphate and the acid-soluble phosphate contents increase regularly from the outside towards the middle. Reheating a basic slag with a low silica content decreases the proportion of acid-soluble phosphate owing to the decomposition of the free calcium tetraphosphate into lime and the less soluble compound, oxyapatite. A. R. POWELL.

Fumigation of stored-product insects with certain alkyl and alkylene formates. R. T. COTTON and R. C. ROARK (Ind. Eng. Chem., 1928, 20, 380–382; cf. B., 1927, 862).—Quantitative laboratory experiments showed that the vapours of methyl, ethyl, propyl, isopropyl, isobutyl, isoamyl, and allyl formates are all toxic to the rice weevil, and have little or no effect on the germination of wheat. Addition of 60–75% by vol. of carbon tetrachloride renders these formates (with the exception of the methyl and ethyl compounds) "free from fire hazard," i.e., the vapour of the mixture heated to 50° with air will not propagate a flame when sparked. Large-scale tests indicated that some of the mixtures appeared promising as fumigants against various stored-product insects. C. T. GIMMINGHAM.

XVII.—SUGARS; STARCHES; GUMS.

Natural alkalinity [of beet juices]. O. SPENGLER and C. BRENDDEL (Z. Ver. deut. Zucker-Ind., 1927, 801–816).—By the natural alkalinity of beet juice is understood the residual alkalinity after treatment with lime and precipitation of this by carbon dioxide, and it represents free potash and soda originally present as salts. The greater part of the alkali metals in fresh juice from sound beets is combined with acids which are precipitable by lime. In carbonatation, therefore, it is not necessary to leave any free lime to supply the requisite final alkalinity, the liberated potash and soda sufficing for this. These are present as hydroxides at the end of the first carbonatation, and should be converted into carbonates, but not into bicarbonates, by the second. In old or damaged beets the alkali metals are combined with acids which are not precipitable by lime, and they remain as neutral salts after carbonatation, so that there is a deficiency of natural alkalinity, and it becomes necessary to leave some free lime after carbonatation to obtain the necessary final alkalinity. To avoid this, since lime alkalinity is particularly undesirable during evaporation, a suitable amount of sodium carbonate may be added to the juice, e.g., before the final carbonatation. As a guide in ascertaining how much to add, the authors describe methods for

determining the "theoretical" and the "practical" residual alkalinity, on filtered juice from the first carbonatation. The "theoretical" residual affinity is the excess of the total alkalinity (to phenolphthalein) over the lime content (determined by soap solution), both being expressed as CaO. Probably, however, the "practical" residual alkalinity will afford a closer estimate of the condition of the juice after the final carbonatation. It is found by neutralising to phenolphthalein with 0.2*N*-hydrochloric acid, then adding an equal volume of 0.2*N*-sodium carbonate, heating in boiling water, filtering from calcium carbonate, cooling, and determining the residual alkalinity with 0.0357*N*-hydrochloric acid. Full working details and precautions are given. Factory experience alone can decide which of these methods is the more useful. J. H. LANE.

Copper-reducing substances contained in beet-roots and diffusion juice. L. BRANCOURT (Bull. Assoc. Chim. Sucr., 1928, 45, 251—254).—In diffusion juice from beets which have been stored or slightly injured by frost or other influences, the copper-reducing power is not a sure measure of the invert-sugar content. Decomposition of cellulosic and protein constituents of the beets is liable to occur, especially in the last units of the diffusion battery, and give rise to reducing substances without a corresponding disappearance of sucrose. These reducing substances differ from invert sugar in that, with proper treatment of the juice, they are not so liable as the latter to give rise to coloured substances on liming and evaporation. In badly frozen beets the decompositions which set in after thawing result in the formation of viscous products. Reducing substances are then apt to occur in widely varying amounts, and on liming they yield soluble calcium compounds. The precipitate formed on carbonatation is thereby reduced in amount and of a character difficult to filter even when considerable amounts of sodium carbonate are used. In such cases the author recommends heating the juice to actual boiling, at least after the first carbonatation, and adding a granular form of calcium carbonate to assist filtration. J. H. LANE.

Drying of sugar products and determination of moisture. D. SIDERSKY (Bull. Assoc. Chim. Sucr., 1928, 45, 247—249).—Heating of samples in a capsule inserted in a tube through which dry air was passed and to which a calcium chloride tube was attached, showed that at 103° constant weight both of sample and of calcium chloride tube could be attained in 2 hrs. with first-product beet sugars. With second and lower beet products the calcium chloride tube reached constant weight in 2½—3 hrs., but the samples underwent a slight further loss (about 0.1%) during the next hour. With raw cane sugars, constancy of weight could not be reached even in 6 hrs., probably owing to slow decomposition of levulose. For exact determinations of moisture heating *in vacuo* below 95° is recommended, although the ordinary method serves well enough for commercial analyses. J. H. LANE.

Electrometric determination of the ash of sugar factory products. O. SPENGLER and F. TÖDT (Z. Ver. deut. Zucker-Ind., 1928, 1—12).—Electrometric ash

determinations on raw sugars are best made on solutions of 5° Brix, for although errors due to small variations in sugar concentration are least in solutions of about 30° Brix, the results obtained at this concentration are liable to deviate much more widely from the results by incineration than those obtained with 5% solutions, the conductivities of the individual salts present being affected to different extents by high concentrations of sugar. In general, the influence of the conductivity of the water used is depressed in presence of the sugar and its salts. Zerban and Sattler (Facts about Sugar, 1926, 1158) found that water of very low conductivity, 3×10^{-6} , exerted its full influence, but water of conductivity 40×10^{-6} contributed only 27.4×10^{-6} to the conductivity of 5% raw cane sugar solutions. The factor *C* representing the ratio between conductivity and ash content (by incineration) was found by Zerban and Sattler to range from 1476 to 2022 for cane sugars of various origin in 5% solutions. For sugars from the same district it was fairly constant, being 1786 on the average for Cuban, and 1560 for British West Indian sugars. For 50 raw beet sugars tested by Kayser (B., 1926, 685) it ranged from 1680 to 1880. According to Lundén (B., 1926, 927) it is lower for beet factory products than for refinery (*i.e.*, affined) beet products; and, in general, as the authors have confirmed, there is an inverse relation between *C* and the quality of beet products. A high value of *C* is associated with a high ratio of organic to inorganic salts, which indicates inferior quality. For raw cane sugars Zerban and Sattler found that if *k* is the specific conductivity of a filtered solution containing 10 g. of sample in 200 c.c., and *k*₁ that for a similar solution containing also 5 c.c. of 0.25*N*-hydrochloric acid, the ash content is given by the formula: $0.0001757(9.13k + 1935 - k_1)$. The results show much more uniform agreement with incineration results than when a single electrometric factor is used. By this formula variations in the amount of organic salts present are largely compensated, since they correspondingly lessen the value of *k*₁ by the replacement of part of the added mineral acid by feebly dissociated organic acids. J. H. LANE.

Applicability of the methods of beet sugar factories to the production of lactose. O. UNGNADE (Chem.-Ztg., 1928, 52, 69—71).—The whey which forms the usual raw material of lactose manufacture contains about 4.83% of lactose, 1.17% of organic non-sugars, and 0.64% of ash. It contains much more acid than raw beet juice, and is not adapted to liming and carbonatation, as calcium salts not precipitable by carbon dioxide would be formed. The protein is best coagulated by heat, the neutralised whey being brought to 90° in steam-heated vessels and kept for ½ hr. Heating by injection of steam is often practised, but causes unnecessary dilution. The clear upper liquid is drawn off for evaporation, and the sludge is passed to filter presses which, under a liquid head of 2—3 m., work very satisfactorily and are much preferable to the bag filters still used in some factories. In evaporation great economy would result from the replacement of the single-unit evaporators now generally employed, by small-scale multiple-effect plant, in which the whey could be safely

brought to a dry substance content of 50%. Crystallisation is usually carried out in water-cooled copper tanks, but a modified form of "malaxeur," as used in sugar factories for crystallisation in motion, gives better results. The purging of the crystals in the centrifuge is best effected by a fine water spray delivered under 3–4 atm. pressure. The raw sugar contains lactose 92.7%, water 1.85%, organic non-sugar 3.34%, and ash 2.14%. The refining of the product could be rendered more efficient by improved conditions of crystallisation and the use of decolorising carbons, sodium hyposulphite, or sulphur dioxide. J. H. LANE.

Clerget factor and the Deerr method of double polarisation. C. E. COATES and C. SHEN (Ind. Eng. Chem., 1928, 20, 70–74).—With Deerr's method for determining sucrose (B., 1915, 503) the authors found inversion constants consistently about 1.5 units lower than those given by Deerr. To avoid possible sources of error they propose certain modifications. The direct polarisation is made on a solution treated only with barium hydroxide and aluminium sulphate solutions in equivalent amounts. The inversion is carried out on a separate portion of the original solution with 0.4*N*-sulphuric acid by heating to 68° in 3 min. and maintaining this temperature for 7 min.; after cooling, the same volume of aluminium sulphate solution is added as was added to the other, and the liquid is exactly neutralised (to rosolic acid) with the barium hydroxide solution, filtered, and read. Corrections are made for the volumes of the precipitates. The inversion constant found for half sugar normal solutions is 141.7, and that for quarter sugar normal solutions 140.89.

J. H. LANE.

Decomposition of sucrose by adsorbent carbons. E. LANDT (Z. Ver. deut. Zucker-Ind., 1927, 834–840).—A mathematical exposition of Vašátko's results (B., 1928, 29.)

J. H. LANE.

Corn starch. STALEY and HELFRECHT.—See XI.

PATENTS.

Diffusion process and apparatus. K. KOMERS and K. ČUKER (B.P. 274,131, 11.7.27. Czechoslov., 10.7.26).—Materials with undamaged cell membranes (e.g., beet slices, sugar cane wood, bark, etc.) are freed mechanically from superficially adhering juices, and then treated with a current of preheated air, flue gases, or other gas, which, without injury to the cell membranes, removes part of the internal moisture of the cells and oxidises the cell conglomerates (the oxidation, which may be done separately, converts leuco-bases and coagulates colloids). In practice, the material, freed from superficial juices before entering the diffuser or between individual elements of a battery of diffusers, is passed through a vessel for treatment with gases. By this process beets may be completely leached out in 25 min.

B. FULLMAN.

Production of starch soluble in cold water. PFEIFFER, and Dr. SCHWANDNER G.M.B.H. (G.P. 445,557, 1.11.24).—Hot water in a finely-divided form is added to dry starch, in quantity sufficient to work it up into a thick paste which only needs drying in the air.

L. A. COLES.

Preparation of [pure] tricalcium saccharate. C. STEFFEN, JUN. (U.S.P. 1,667,446, 24.4.28. Appl., 27.4.26. Austr., 17.11.25).—See B.P. 261,693; B., 1927, 454.

Salts from fermented molasses (B.P. 288,390).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Sarcinæ. K. SILBERNAGEL (Woch. Brau., 1928, 45, 143–148, 155–160, 165–168).—Sarcinæ were isolated on nutrient agar from beer and from air and classified into two groups. The yellow varieties in both the packet and non-packet forms exhibited a uniform and inherently related behaviour with regard to growth and other properties, whilst the colourless species, differing both from the yellow variety and also among themselves, showed marked individuality. Two of the most suitable sources of carbon for the reproduction of the micro-organism were lactic acid and pyrotartaric acid, whilst, of the carbohydrates, achroodextrin, inulin, and lævulose were assimilated most easily. Lactic acid had a specially stimulating action, and a few drops of its solution, when added to the nutrient, produced a rapid development of the organism. The nitrogen requirements of the sarcinæ were best satisfied by ammonium salts, and their presence produced rapid reproduction. Urea was assimilated very easily, but peptone induced a lag period prior to later development. The ease with which amide-nitrogen was assimilated depended on the complexity of the compound. Rapid growth accompanied the use of alanine, whereas the nitrogen from asparagine and leucine was assimilated with difficulty. Glycine was not so effective a source of nitrogen as alanine, whilst methylamine, nitrates, and nitrites yielded no nitrogen. Among the enzymes present in the sarcinæ were amylase, lipase, and the proteolytic enzymes in moderate amounts. Catalase and peroxidase were found in greater quantity, whilst invertase and amidase occurred as traces. Of the other enzymes tested, maltase, inulase, and lactase were absent.

C. RANKEN.

Taka-invertase. R. WEIDENAGEN (Z. Ver. deut. Zucker-Ind., 1928, 125–134).—Taka-diaxase was found to be without action on melibiose, but to hydrolyse raffinose and sucrose at velocities in the ratio of 1:1.96. It is concluded that taka-invertase is a fructo-invertase and is not associated with a melibiase as supposed by Leibowitz and Mechlinski (A., 1926, 865). The decomposition of raffinose was complete within 24 hrs., and even after 96 hrs. no carbohydrates other than melibiose and lævulose could be detected. The optimum p_H value for taka-invertase is about 5.0. Evidence for and against the existence of gluco- and fructo-invertases is considered.

F. E. DAY.

Evaluation of a dried yeast in relation to its biological action. W. WEICHARDT and H. UNGER (Pharm. Ztg., 1928, 73, 526–527).—A quantitative examination of the biological value of "Levurinos," a commercial dried yeast.

E. H. SHARPLES.

English barleys of 1927. H. M. CHUBB (J. Inst. Brew., 1928, 34, 214–217).—Owing to the very wet

season about 30% of the barley crop was unfit for malting. The general average of the available crop was poor, but the best barleys malted well. Local conditions and environment were greater factors than usual, and good barleys were found in small quantities all over the country, the best being on the edge of the chalk in West Norfolk, Suffolk, and East Cambridge.

C. RANKEN.

Critical analysis of malt. A. T. HENLEY (J. Inst. Brew., 1928, 34, 223—225).—Samples of malt are separated into fractions of differing sp. gr. by flotation liquids which are prepared by mixing benzol and carbon tetrachloride. Both of these liquids are substantially without effect on the malt, and can be removed easily by a current of dry warm air. Examination of the quantity of extract obtained from each of the several fractions allows information to be drawn as to the reasons for any want of modification of the malt, whilst any trace of overheating in the kiln will be reflected almost entirely in the analytical values obtained from only one of the fractions.

C. RANKEN.

Finings. H. W. HARMAN, J. H. OLIVER, and P. WOODHOUSE (J. Inst. Brew., 1928, 34, 203—213).—In the absence of other colloids, maximum precipitation of the finings occurs in the neighbourhood of an isoelectric point of p_H 5.0, approximately, whilst with low rates of addition no precipitation takes place at p_H 3.7 and p_H 4.0. From a comparison of the result of fining beer and buffered solutions it is concluded that fining takes place chiefly owing to the mutual precipitation effect produced when two colloidal solutions of electrically opposite charges are mixed. A certain amount of finings are left in solution, which stabilises the other colloids in solution. This protective power varies according as the method of cutting the isinglass, and is entirely absent in finings which have been overcut. The action of finings may be retarded by the presence of wild yeast, excessive hop rates, and by variation of the temperature during fining, whilst the presence of carbon dioxide accelerates the action. Irish moss acts as a precipitant to isinglass fining.

C. RANKEN.

Samsu from rice. R. O. BISHOP and G. L. TEIK (Malay. Agric. J., 1928, 16, 14—19).—The process used by the Chinese in Malaya depends on the utilisation of a mould containing a ferment (*Amylomyces Rouxii*) which converts moist starch into alcohol. The mould is added in the form of "rice-cake" consisting of rice starch, soya beans, clay, vegetable tissue, and variable amounts of mucors and yeasts. The rice is boiled, allowed to cool, and the rice cake roughly mixed into it in the proportion of 3 pts. of rice to 1 pt. of rice cake. The mixture is placed in earthenware jars with water and left for 27 days. Distillation is carried out in a primitive apparatus which is described. A yield approximately 54% of the theoretical is obtained. This could be considerably increased if suggested improvements in the cooking, fermentation, and distillation were introduced; such changes would, however, probably alter the aroma and flavour of the beverage.

W. J. BOYD.

Uniform behaviour of bottom-fermentation beer

yeast in respect of fermentation, reproduction, and acid-formation, on storage under water at various temperatures. F. STOCKHAUSEN and F. WINDISCH (Woch. Brau., 1927, 44, 557—564, 573—579).—Similar experiments to the earlier ones (cf. B., 1928, 31) were made with three other typical bottom yeasts, and with the same results.

J. II. LANE.

Sugar : alcohol ratio and the stability of sweet wines. P. MALVEZIN (Bull. Assoc. Chim. Sucr., 1928, 45, 396—399).—In connexion with his work on the preparation of vaccines for the prevention of secondary fermentation, the author noted that he was always successful when the sugar/alcohol ratio was less than 3, but never when the ratio exceeded 4. On reference to the data for over 100 samples of sweet wines received in his laboratory on account of secondary fermentation, all were found to give a ratio of over 4 and generally nearer 6. Samples which had been recorded as keeping well had mean sugar/alcohol ratios of: Gironde 1.3, Sauterne 3.65, Anjou 3.81, Gaillac 7.31. The author concludes that for sweet wines generally, the ratio should not exceed 3.5, but may reach 3.8 in Anjou wines, and 7 in Gaillac and similar wines in which secondary fermentation is usual.

F. E. DAY.

PATENTS.

Clarification of beer, vinegar, and other like liquids. A. J. MURPHY, and MURPHY & SON, LTD. (B.P. 286,861, 18.2.27).—Finings for the clarification of beer, vinegar, and other liquids are produced by covering a suitable quantity of isinglass with water to which is added lactic acid in suitable proportion. As the isinglass progressively swells under treatment, more water is added and the mass stirred, churned, or sieved. Phosphoric acid or other suitable mineral or organic acid may be used in addition to the lactic acid.

C. RANKEN.

Collection of inorganic potassium salt, betaine salt, and glutamic acid from the waste liquor produced in distilling alcohol from fermented beet molasses. Y. TAKAYAMA (B.P. 288,390, 22.1.27. Cf. B.P. 233,196; B., 1925, 568).—The waste liquor is electrically or otherwise dialysed at 40—70°, yeast, proteins, etc. being thus removed. The dialysed liquor is concentrated, treated with hydrochloric acid, kept at 100° for 1 hr., and cooled, when potassium chloride separates. Alternatively, the concentrated liquor is treated with chamber sulphuric acid, heated, and, after removal of potassium sulphate, slaked lime is added to remove excess acid. On concentrating the mother-liquor, betaine hydrochloride (containing potassium chloride) crystallises. The residual liquor is treated with hydrochloric acid and heated under pressure at 110—120° for 1—2 hrs. to convert glutimic into glutamic acid. On concentration, crude glutamic acid hydrochloride crystallises, and is purified by recrystallisation or conversion into calcium glutamate. B. FULLMAN.

Extraction of glycerin from distillery vinasses. SOC. DES ÉTABL. BARBET (B.P. 274,519, 19.7.27. Fr., 19.7.26).—Petroleum, toluol, benzol, or other like product is added to the concentrated distillation vinasses and the mixture superheated under a pressure of 6—8 kg.

The pressure is suddenly lowered and self-evaporation produced, by means of which part of the glycerin is carried over with all the petroleum and part of the water. The first portion of the distillate is later added to the main bulk of the vinasses for re-treatment, whilst the second portion, after rectification, is purified by charcoal, filtered, and concentrated in a vacuum. C. RANKEN.

Recovery of nitrogen and acetone from vinasses and/or molasses. NOUVELLES INDUSTRIES CHIMIQUES Soc. ANON. (B.P. 287,829, 13.6.27. Addn. to B.P. 277,932; B., 1928, 345).—An alkaline-earth carbonate, or residue from a previous distillation, or a hygroscopic substance, *e.g.*, sawdust, is added to the mixture of vinasses or molasses and lime and subjected to destructive distillation up to 350°. H. ROYAL-DAWSON.

Production of citric acid. DISTILLERS CO., LTD., and A. M. PEAKE (B.P. 287,604, 27.11.26).—Fruit pulp, wood dextrins, gums, and similar pectinous substances are treated with citric acid enzymes present in the pith of fresh and preferably unripe citrus fruit from which the essential oils have been removed. A small amount of manganese or similar oxygen-carrying catalyst may be added. The reaction should be carried out at 25–35°, and moderate aeration is advantageous.

C. RANKEN.

Filters [for wines and liqueurs]. J. CUÉNOUD (B.P. 288,920, 8.11.27).

XIX.—FOODS.

Use of Janus-green in the reductase test for milk. A. R. TANKARD (Analyst, 1928, 53, 213).—A comparison of results given in the reductase test by 35 samples of commercial milk using both Janus-green and methylene-blue gave similar results. The rising of the cream in the tubes appears to inhibit oxidation. Janus-green acts more slowly than methylene-blue, and the red colour produced in the first stage of the reduction shows early in the cream when the dye is about to be reduced throughout the milk, and in some instances may show when the full reaction does not occur within the time limit. A bacterial count shows the number of organisms to be generally lower than that indicated by either form of the test.

D. G. HEWER.

Determination of salt in margarine. D. W. STEUART (Analyst, 1928, 53, 212–213; cf. B., 1928, 241).—If to 3 g. of melted margarine 10 c.c. of acetone and a few drops of chromate indicator are added, and the mixture is titrated with 0.1*N*-silver nitrate solution, the same result is obtained as if the aqueous extract of the solids-not-fat is used.

D. G. HEWER.

By-products of the pineapple canning industry. V. R. GREENSTREET and G. L. TEIK (Malay. Agric. J., 1928, 16, 8–13).—Malayan methods of pineapple canning are inefficient compared with those employed in Hawaii, owing to the uneconomic disposal of waste matter. This material, consisting of cores, skins, and rotten and unripe fruit, is in the proportion of 2 pts. to each part of canned fruit. It contains about 90% of moisture, together with fibre, pentoses, sugars, organic acids and protein matter. Possible methods of disposal

are: (a) incineration to produce a valuable manure (the ash contains about 6% P_2O_5 and 24% K_2O); (b) conversion into cattle food; (c) spreading the crushed waste in shallow layers on the ground so that it decomposes. All such methods involve a preliminary grinding and pressing whereby a liquid containing sugars is produced, disposal of which must also be considered. In Hawaii the waste is converted into "pineapple bran"—a valuable cattle food—containing water 10.63%, protein 3.62%, ether extract 1.01%, invert-sugar 11.96%, starch 42.15%, fibre 18.23%, and ash 3.70%. Trials showed that the fermentation and distillation of the juice, for the production of a potable alcoholic liquor similar to brandy or samsu, can be carried out without difficulty.

W. J. BOYD.

Aluminium vessels. BUSCHLINGER.—See I. **Ultra-violet light through glass.** STARKIE and TURNER.—See VIII.

PATENTS.

Treatment of cereals. TREUHAND-GES.M.B.H. BARTMANN & Co. (B.P. 270,706, 2.5.27. Ger., 6.5.26).—The grains are steeped in water containing formaldehyde or lime to cause softening and swelling of the endosperm, during which intramolecular respiration is prevented by supplying oxygen to the grains by introduction of air or oxygen into the steeping liquor, by temporarily lifting the grains out of the liquor, or by transferring the grains from one steeping bath to another.

F. R. ENNOS.

Preparation of vegetable food. E. H. MILES and G. REILLY (B.P. 274,051, 17.6.27. U.S., 8.7.26. Addn. to B.P. 256,765).—The juices extracted from vegetable material having essentially dietetic properties (*e.g.*, lettuce, cabbage, etc.), and from other vegetable material having also preservative properties (*e.g.*, carrots), are heated separately to 80°, cooled, and strained, then mixed together, again heated to 80° until hydrolysis of the sugars is well advanced, cooled to about 30°, and concentrated *in vacuo*, the total time of heating not exceeding 1 hr.

L. A. COLES.

Manufacture of live-stock feeds. U.S. FARM FEED CORP., Assees. of C. R. MABEE (B.P. 272,447, 7.3.27. U.S., 11.6.26).—Farm roughage is loosely packed in a wire-mesh container and moistened either with water or with a solution containing a fermentative agent and other material, *e.g.*, malt with calcium and magnesium hydroxides, sulphur, and sodium chloride. It is then allowed to ferment for one or two days in a slowly moving current of air, which is made to circulate through the material by the application of heat or by utilisation of the heat of fermentation.

F. R. ENNOS.

Purification of edible oils (Addn. F.P. 31,181).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Tests for impurities in ether. I. Test for peroxides. G. MIDDLETON and F. C. HYMAS (Analyst, 1928, 53, 201–209).—The relative sensitiveness of a number of different tests both for hydrogen peroxide and ether peroxide in ether was determined, the most

useful being potassium iodide and starch, reduced phenolphthalein, leuco-methyl-green and peroxidase, and ferrous thiocyanate. If the maximum attainable purity is to be enforced the ferrous thiocyanate test should be used. It gives uniformly consistent results, and blank tests are readily obtained with pure ether. The amount of peroxide permissible might be defined by comparing the colour of the ether layer, after remaining for 5 min., either with a standard solution of cobalt sulphate or with tintometer glasses. In the former case the tint should not be greater than that of an equal depth of an aqueous solution containing 0.15 g. of crystalline cobalt sulphate and 2 c.c. of dilute sulphuric acid in 100 c.c. (equal to 0.3 red unit on the Lovibond scale for a depth of 30 mm. of liquid). The ferrous thiocyanate solution must be colourless, and may be freshly reduced by a dilute solution of titanous chloride (avoiding any excess of this reagent), or prepared from metallic iron, sulphuric acid, and potassium thiocyanate in an atmosphere of carbon dioxide, in which case it may be kept for several weeks if air is completely excluded. The usual pharmacopœia tests are regarded as unsatisfactory. D. G. HEWER.

Standardisation of ephedrine and its salts.

J. B. PETERSON (Ind. Eng. Chem., 1928, 20, 388—391).—Standards are proposed for the control of the purity of ephedrine and its hydrochloride and sulphate. Ephedrine hydrochloride is soluble in chloroform, has m.p. 216—220°, $[\alpha]_D^{20} = 33.0^\circ$ to -35.5° , whilst ephedrine sulphate has m.p. 240—243° (corr.; the rate of heating being strictly according to the method of U.S.P. X.), $[\alpha]_D^{20} = 29^\circ$ to -30° . On treating an aqueous solution of the sulphate with ammonia water, extracting with chloroform, and evaporating the chloroform extract, crystals of ephedrine hydrochloride are obtained, but the reaction is not quantitative as benzaldehyde is formed as a by-product; the hydrochloride may also be produced when a chloroform solution of ephedrine is evaporated. Ephedrine has m.p. 34—40°, b.p. above 200°, and $[\alpha]_D^{20} = 6.0^\circ$ to -7.5° . With copper sulphate and sodium hydroxide solution ephedrine and its salts yield a reddish-purple coloration which is partially removed on addition of ether. Crystallographic data are given for the hydrochloride and sulphate. W. J. POWELL.

Determination of yield of "digitaline cristallisée" [obtainable from *Digitalis* leaves]. E. PERROT and P. BOURCET (Compt. rend., 1928, 186, 1021—1024).—A method is described by which the yield of commercial "digitaline cristallisée" (Kiliani's digitoxin) from *Digitalis* leaves may be determined. The alcoholic extract of the powdered leaves is treated with basic lead acetate, evaporated, and the residue extracted with cold chloroform. After evaporation of the chloroform and extraction of the residue with pinene and ether, the solid is again extracted with chloroform and the "digitaline" precipitated by addition of ether and light petroleum. R. K. CALLOW.

Detection of colocynthin in colocynth extract. L. DÁVID (Pharm. Ztg., 1928, 73, 525—526).—The isolation of colocynthin, colocynthidin resin, and colouring matter from colocynth extract is described. In the Keller colour reaction only colocynthin gives the crim-

son-coloured ring, and with the exception of the colouring matter, which forms a brown ring, the other constituents give no colour. The test has been modified as follows: 0.2 g. of the extract containing dextrin is mixed with lime paste to the size of a small bean and the mixture is dried on the water-bath. The pulverised residue is extracted twice with 1 c.c. of methyl alcohol each time, collected on a parchment filter moistened with alcohol, and the filtrate evaporated. The residue is dissolved in 2 c.c. of glacial acetic acid, one drop of ferric chloride solution is added, and 2 c.c. of concentrated sulphuric acid are run carefully down the side of the tube. A vivid, carmine-red ring is formed at the junction of the two liquids. The test is reliable and an extract containing only 2% of colocynthin gives a pale but quite perceptible colour. E. H. SHARPLES.

West Australian sandalwood oil. P. MAY (Pharm. J., 1928, 120, 368—369; cf. Perrot, B., 1928, 210).—The physical and chemical constants of 12 West Australian and Mysore sandalwood oils are compared and the alcoholic constituents of a "Plaimar" oil have been examined. West Australian sandalwood oil is now obtainable of good standard quality closely resembling the East Indian oil, to which it is equal in therapeutic value. The properties of the alcohols ($d_4^{15} 0.972$, $\alpha = 9.44^\circ$, $n_D^{25} 1.506$) are not inconsistent with those of a mixture of α - and β -santalols, but are quite incompatible with the fusanols of Rao and Sudborough (B., 1923, 575 A).

E. H. SHARPLES.

Essential oil from *Agathis australis*. J. R. HOSKING (Rec. trav. chim., 1928, 47, 578—584).—The oil obtained in 0.16% yield by steam distillation of the leaves and shoots of *Agathis australis* (kauri pine) during December–February has $d_4^{25} 0.9340$, $n_D^{25} 1.4952$, $[\alpha]_{D_{161}}^{25} + 24.41^\circ$, acid value 0.39. The chief constituents of the oil are citronellal (0.2%), d - α -pinene (64%), d -camphene (0.7%), d -limonene and dipentene (1%), cineole (0.6%), d -borneol (3.2%), bornyl acetate (1.4%), l -cadinene (7%), and a *diterpene* (13%) to which the name *kaurene* is given. Kaurene, probably $C_{20}H_{32}$, contains one double linking and has m.p. 57—58°, $d_4^{20} 0.9631$, $n_D^{20} 1.5132$, $[\alpha]_{D_{161}}^{25} \pm 0^\circ$, when regenerated from its hydrochloride, m.p. 110—111°. The oil obtained during June from the leaves of *A. australis* has $d_4^{25} 0.9210$, $n_D^{25} 1.4783$, $[\alpha]_{D_{161}}^{25} + 33.5^\circ$, and contains d - α -pinene (75%), kaurene (6%), and l -cadinene (3.5%).

H. BURTON.

Determination of carvone in dill oil. J. REILLY and P. J. DRUMM (Analyst, 1928, 53, 209—211).—10 g. of dill oil in 120 c.c. of alcohol are added to a cooled solution of 6 g. of semicarbazide hydrochloride in 15 c.c. of water, followed by a solution of fused sodium acetate in 10 c.c. of hot water; after 24 hrs. the semicarbazone crystallises. Water (840 c.c.) is now added and, after keeping, the precipitated carbazone is collected and dried. Values obtained by this method agree closely with those by the neutral sulphite method, and are approximately 4% less than by the oxime titration method. D. G. HEWER.

Determination of total geraniol content of citronella oil. A. RECLAIRE and D. B. SPOELSTRA (Perf. Ess. Oil Rec., 1928, 19, 143).—In the determination of citron-

ellal by the Kjeldahl analysis of the oxime the factor previously given (B., 1927, 427) is not accurate for preparations containing a high percentage of citronellal. An exact formula is given and also a table for use with oils containing 28—55% of citronellal.

E. H. SHARPLES.

Cod-liver oil. DICKHART. **Ergot oil.** BAUGHMAN and JAMIESON.—See XII.

PATENTS.

Manufacture of thymol from umbellulone. E. K. NELSON, Assr. to U.S.A. (U.S.P., 1,666,342, 17.4.28. Appl., 27.9.27).—Thymol is obtained by passing the vapour of California laurel oil or umbellulone through a tube at 280°, being separated from the crude product in the former case by fractional distillation and in the latter by treatment with caustic alkali. B. FULLMAN.

Manufacture of ketones. (Sir) W. J. POPE (B.P. 287,967, 31.12.26).—The monoacyl derivatives of quinol and resorcinol are isomerised by heating with chloride of magnesium, zinc, aluminium, iron, or tin, or sulphuric or phosphoric acid, giving dihydroxyphenyl alkyl ketones, which may be reduced by the usual method to alkylresorcinols. The following compounds are described: *p*-hydroxyphenyl propionate; 2:5-dihydroxyphenyl ethyl ketone; *m*-hydroxyphenyl isobutyrate, *n*-hexoate, b.p. 200—210°/15 mm., laurate, and stearate; 2:4-dihydroxyphenyl isopropyl ketone; 4-isobutylresorcinol, m.p. 62—63.5°; 2:4-dihydroxyphenyl amyl ketone, m.p. 56—57°; 2:4-dihydroxyphenyl undecyl ketone, m.p. 75—76°; 2:4-dihydroxyphenyl heptadecyl ketone, m.p. 90—91°. The ketones and the alkylresorcinols are bactericides. C. HOLLINS.

Manufacture of new pharmaceutical compounds [alkoxymethyltheobromines]. I. G. FARBENIND. A.-G., K. SCHIRANZ, and C. LUTTER (B.P. 288,366, 6.1.27. Addn. to B.P. 242,296; B., 1926, 216).—Theobromine (or a metal salt) is treated with a chloromethyl alkyl ether, prepared from formaldehyde, hydrogen chloride, and a homologue of methyl alcohol, to give 3:7-dimethyl-1-alkoxymethylxanthines. These, unlike the 1-methoxymethyl compound, have no action on the heart, but are diuretics and give neutral solutions in aqueous sodium salicylate suitable for injection. 1-Ethoxy-methyl- (m.p. 152—153°), 1-isopropoxymethyl- (m.p. 107—108°), 1-*n*-propoxymethyl- (m.p. 130—132°), 1-*n*-butoxymethyl- (m.p. 102—103°), 1-isobutoxymethyl- (m.p. 128—129°), 1-benzoyloxymethyl- (m.p. 95—97°), 1-allyloxymethoxy- (m.p. 108—110°) derivatives are described; also chloromethyl *n*-butyl ether, b.p. 129—136°/15 mm., benzyl chloromethyl ether, b.p. 105—117°/15 mm., and allyl chloromethyl ether, b.p. 100—110°/15 mm. C. HOLLINS.

Preparation of addition compounds of deoxycholic acid possessing bactericidal properties. F. W. R. OSTEN (B.P. 287,965, 30.12.26).—Deoxycholic acid and its alkali salts form with safranine and acridine dyes non-toxic, bactericidal, and trypanocidal additive products. These dissolve insoluble therapeutic substances, e.g., quinine. The deoxycholic acid-tolusafranine additive product is described.

B. FULLMAN.

Manufacture of disodium salts of sulphomethyl-aminometalmercaptosulphonic acids. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 285,001, 9.8.27. Ger., 8.2.27. Addn. to B.P. 270,729; B., 1927, 860).—The formaldehyde-bisulphite compounds of 3-amino-4-aurothiolbenzenesulphonic acid, 4-(3'-amino-2'-pyridyl)-amino-2-aurothiolbenzenesulphonic acid (from 3-aminothiophenol-6-sulphonic acid and 2-chloro-3-aminopyridine and subsequent reduction and introduction of gold), and 3-amino-4-argenthiothiolbenzenesulphonic acid, are described. C. HOLLINS.

Manufacture of a new syphilis remedy from diaminodihydroxyarsenobenzenemonomethylene-sulphonic acid. T. IWADARE (U.S.P. 1,665,787, 10.4.28. Appl., 8.10.26. Japan, 14.12.25).—Diaminodihydroxyarsenobenzenemonomethylenesulphonic acid is treated with formaldehyde and sodium hydrogen sulphite. B. FULLMAN.

[Organic] arsenical compound. C. S. HAMILTON, Assr. to PARKE, DAVIS & Co. (U.S.P. 1,665,781, 10.4.28. Appl., 17.3.24).—A group containing hydroxyl is introduced into the amino-group of arsanilic acid derivatives by treating them with halogenohydrins.

B. FULLMAN.

Manufacture of new complex antimony compounds. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 288,370, 7.1.27. Addn. to B.P. 271,940; B., 1927, 573).—Antimony compounds of polyhydric phenols containing two *ortho*-hydroxyl groups are combined with neutral salts of carboxylated or sulphonated derivatives of the same or similar phenols, e.g., salts of gallic acid, carboxygallic acid, pyrocatechdisulphonic acid, and 2:3-dihydroxynaphthalenedisulphonic acid. Examples are antimonypyrocatechol with sodium gallate and with sodium pyrocatecholdisulphonate. The products are non-irritant trypanocides. C. HOLLINS.

Manufacture of farnesol. L. RUZICKA, Assr. to M. NAEF & Co. (U.S.P. 1,663,817—8, 27.3.28. Appl., [A] 19.3.24, [B] 2.10.25. Switz., 22.3.23).—See B.P. 213,251; B., 1925, 691.

Production of diacylisoithiocarbamide ether. H. SCHOTTE, Assr. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,667,053, 24.4.28. Appl., 12.7.26. Ger., 16.7.25).—See B.P. 255,466; B., 1927, 172.

Preparation of organic arsenic compounds. A. BINZ and C. RÄTH (U.S.P. 1,667,237, 24.4.28. Appl., 13.7.26. Ger., 22.7.25).—See B.P. 255,839; B., 1927, 829.

Reduction of arsinic acids. A. BINZ and C. RÄTH (U.S.P. 1,667,238, 24.4.28. Appl., 13.7.26. Ger., 22.7.25).—See B.P. 255,892; B., 1928, 36.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Mordanting of basic dyes. MICHELS.—See VI.

PATENTS.

Production of gelatin relief pictures. S. PROCOUDINE GORSKY & CIE. [SOC. DE PHOTOCHIMIE, "ELKA"] (G.P. 445,513, 19.12.24).—Films comprising layers of gelatin and silver bromide, prepared separately in the

usual manner from pure gelatin and silver bromide practically free from colloids, are exposed to light, developed with pyrogallol or pyrocatechol, and washed.

L. A. COLES.

XXII.—EXPLOSIVES; MATCHES.

Flammability of refrigerants. Mixtures of methyl and ethyl chlorides and bromides. G. W. JONES (Ind. Eng. Chem., 1928, 20, 367—370).—With increasing depth of mines the use of refrigerants may be necessary, and such should preferably be non-flammable. The author's tests were carried out in a bell-jar resting in another bell inverted and containing mercury as a seal. A gas connexion, mixing fan, and electrodes were fitted. A mixture was considered to have propagated if the flame travelled to the top of the bell-jar and spread. The flammable limits for mixtures with air were found to be as follows: methyl chloride 8.25—18.70%, ethyl chloride 4.00—14.80%, methyl bromide 13.50—14.50%, and ethyl bromide 6.75—11.25%. Mixtures were found to follow Le Chatelier's law approximately (cf. Coward and others, B., 1919, 126 A), and no mixture was non-flammable. These compounds are more easily ignited by an intense electric spark than by a flame, with which the bromides are in fact non-flammable. Analysis of the mixtures was performed by diluting with a mixture of equal volumes of oxygen and air, adding gases from electrolysis of water to render the mixture thoroughly inflammable, and measuring the contraction on explosion and on absorption of carbon dioxide.

C. IRWIN.

PATENT.

Gelatin-dynamite explosive. R. B. SMITH and E. M. SYMMES, Assr. to HERCULES POWDER Co. (U.S.P. 1,667,083, 24.4.28. Appl., 27.11.26).—A low-strength dynamite explosive is composed of 17—18% of nitroglycerin, 2—4% of dinitrotoluol, 0.1—0.2% of nitro-cotton, 60—65% of sodium nitrate, 9—12% of sulphur, 2—5% of starch, and a little chalk. F. G. CROSSE.

XXIII.—SANITATION; WATER PURIFICATION.

Disinfectant action of sodium toluene-*p*-sulphochloroamide, particularly of "Chloramine-Heyden." R. DIETZEL and F. SCHLEMMER (Arch. Pharm., 1928, 266, 173—188).—"Chloramine-Heyden," together with several other brands of "chloramine-T" has been examined for bactericidal action on staphylococci organisms by Paul and Krönig's method (Z. Hyg. Infekt., 1897, 25, 1). It is superior to phenol, but much inferior to mercuric chloride. It is also not so active as inorganic hypochlorites, but here the action is dependent to some extent on the free alkali present. The results obtained by Paul and Krönig's method are not directly comparable with those obtained in a different way.

W. A. SILVESTER.

Carbon disulphide in viscose manufacture. J. EGGERT (Chem.-Ztg., 1928, 52, 289).—The importance of the protection of workers in factories where carbon disulphide is used is pointed out. The liquid, besides

being very inflammable, acts as a poison when breathed and when allowed to come into contact with the skin. The action on the skin appears to be connected with the dissolving of surface grease and subsequent attack of the layer of fat just beneath the skin. Various symptoms of the poisoning are given, and suggestions are made for reducing the contact of the workers with the liquid or vapour to a minimum.

H. INGLESON.

Iron and manganese in water. H. THIELE (Gas-u. Wasserfach, 1928, 71, 289—290).—Processes for the removal of iron and manganese from water depend usually on the negligible solubility of ferric and manganic hydroxides as compared with the appreciable solubility of ferrous and manganous hydroxides. Oxidation of the latter by the air in a suitable filter is catalysed by the layer of ferric or manganic hydroxide first deposited on the contact material used, and also by the presence of certain micro-organisms. Manganese is often removed by the use of the dioxide as oxidising agent, in the form of pyrolusite, precipitated on coke, brick, etc., or in the form of manganese-permutit. Active charcoal will remove both iron and manganese, but its activity in that respect rapidly diminishes.

A. B. MANNING.

Determination of plankton [in water]. W. F. LANGELEI (J. Amer. Water Works' Assoc., 1928, 19, 408—415).—For laboratory work the Sedgwick-Rafter method, described in the A.P.H.A. Standard Methods of Water Analysis, has many advantages, but the method of counting is tedious. In routine examination of water supplies, with the object of controlling the quantity of plankton present, Henson's net method is preferable. The nets used are conical with a copper or glass container. The volume of plankton collected is measured after settling or centrifuging and the results are quoted in c.c./m.³ The main errors of this method depend on the velocity of haul and the ratio of net surface to area of circle. Certain small plankton pass through the net, but these have seldom been shown to have a deleterious effect on taste or odour. Their presence is often indicated by a loss of transparency by the water, and this is used to indicate the need for copper sulphate treatment.

C. JEPSON.

Fumigants. COTTON AND ROARK.—See XVI.

PATENTS.

Production of fumigating mixtures containing hydrocyanic acid. DEUTS. GOLD- U. SILBER-SCHNEIDEN-STALT, VORM. ROESSLER (B.P. 271,514, 23.5.27. Ger., 22.5.26).—Mixtures of hydrocyanic acid and cyanogen chloride are prepared by the fairly rapid passage of chlorine into cooled liquid or concentrated (stronger than 92%) hydrocyanic acid, in the presence of non-alkaline substances (e.g., oxides, hydroxides, carbonates, etc.), which may also be added subsequently, to remove the hydrochloric acid formed.

B. FULLMAN.

Plant for dealing with sewage. PULSOMETER ENGINEERING Co., LTD., and J. BJÖRNSTAD (B.P. 288,814, 11.4.27).

Apparatus for treating liquids [water] with zeolites. H. BARON. From W. NEUMANN (B.P. 288,660, 5.11.26, 25.4 and 1.7.27).

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JUNE 22, 1928.

I.—GENERAL; PLANT; MACHINERY.

Goldberg spectrodensograph. H. SCHERING (Collegium, 1928, 143—150).—An improved apparatus enables an unskilled operator to obtain in a few minutes graphs showing the colour density between the wavelengths 700—400 μ . The graph is plotted automatically. Non-transparent materials, *e.g.*, leather, paper, fabrics, as well as transparent coloured materials can be examined. The absorption curves are thus obtained without difficulty. The apparatus comprises a monochromator side by side with a photometer. D. WOODROFFE.

Pendulum viscosimeter. ALBRECHT and WOLFF (Petroleum, 1928, 24, 551—555).—The apparatus described is suitable for the rapid determination of the viscosity of any liquid at temperatures up to 300°; 200 c.c. only of the liquid are required. The apparatus consists of a pendulum, part of which is immersed in the liquid to be tested. A mirror is attached to the pendulum at its point of suspension and reflects a ray of light from a fixed source upon a moving photographic film. The liquid is brought to the required temperature, and the pendulum is displaced through a constant angle from its mean position and set in vibration. The oscillations are recorded upon the moving film by the ray reflected from the mirror. The theory of the method is discussed, and an equation for the instrument arrived at, from which the viscosity of the liquid is obtained.

R. C. ODAMS.

Simple method for determining the absolute viscosity of oils. P. E. RAASCHOU (Dansk Tidsskr. Farm., 1928, 2, 134—139).—The apparatus consists of a glass capillary tube with three marks at suitable intervals, fitted into a test tube by means of a stopper, the whole being immersed in a container filled with water. The oil is introduced into the inner test tube, and when the temperature has become constant (15 min.) a little is drawn up the capillary tube to a point above the upper mark, a rubber ball being affixed to the capillary for this purpose. After releasing the ball, the time required for the level of the liquid in the capillary to fall from the upper to the middle mark is accurately noted. The viscosity is then given by the formula $\eta = kt$, where k is a constant for the apparatus, which is found by employing liquids of known viscosity, preferably paraffin or a solution of glycerin. The temperature must be kept constant, and a correction is necessary if the density of the liquid to be determined differs greatly from that used in calibrating the apparatus.

H. F. HARWOOD.

Determination of specific gravity of powders. H. RASQUIN (Farben-Ztg., 1928, 33, 1786—1787).—The

specific gravity of powders insoluble in and heavier than water is determined by introducing 10 g. of the substance through a funnel into 10 c.c. of water in a measuring cylinder, the exit tube of the funnel being kept just clear of the water. The specific gravity is found by dividing 10 by the increase in volume in c.c.

Design of fractionating columns. D. B. KEYES, R. SOUKUP, and W. A. NICHOLS, JUN. (Ind. Eng. Chem. 1928, 20, 464—466).—The graphical determination of the minimum number of plates necessary for a fractionating column for a given duty is effected as follows. A curve is drawn showing the composition relation between the vapour and the boiling liquid; the equal-composition curve (a straight line at an angle of 45°) is drawn between 0 and 100% composition of the more volatile compound; and the compositions of the residue, the feed, and the distillate are drawn as vertical straight lines. Then the number of steps between the two curves from the distillate line to the residue line is counted, and a safety factor to provide for reasonable reflux is applied. Several examples of the calculation are given.

C. IRWIN.

Manometer for vacuum distillation. F. FRIEDRICH (Chem.-Ztg., 1928, 52, 272).—A manometer which is independent of atmospheric pressure and can readily be filled with mercury and freed completely from gas and moisture comprises the usual open and closed parallel tubes enclosed in an outer closed protecting tube, and connected to each other through a small valve on the injector principle. To fill the manometer mercury is poured into the open tube, and the closed tube is heated in an inclined position until the air is displaced and the mercury boils gently. The manometer is then filled with mercury until the metal reaches to the cylindrical part of the open tube, when the apparatus is ready for use.

A. R. POWELL.

Revised psychrometric chart assists high-temperature design [of dryers]. I. LAVINE and R. L. SUTHERLAND (Chem. Met. Eng., 1928, 35, 224—228).—The chart, which is given, comprises a series of curves showing the relation of absolute humidity, latent heat, specific volume, and other humidity data against temperatures from 40° F. to 700° F., this scale being used. In addition, the "humid heat" (heat required to raise 1 lb. of air 1° F. with its accompanying water vapour) is plotted against the absolute humidity, and a series of adiabatic cooling curves is given. The conditions in a dryer are practically adiabatic. An example is given of the use of the chart in determining the conditions of the exit air from a direct-contact coal dryer, the temperatures and the humidity of the entering air being known.

C. IRWIN.

Building of containers for severe service. T. McL. JASPER (Ind. Eng. Chem., 1928, 20, 466—470).—The building of containers for severe conditions involves a knowledge on the part of the builder of the service conditions proposed, the proper design and protection of the vessel during building, and a suitable test. The strength of steels at high temperatures can only be obtained by long-period tests. These may be carried out in molten lead, the stress-breaking time curve being plotted. Corrosion is greatly increased in many cases by increase of stress or increase of pressure with equal stress. Full-scale tests are desirable and have shown the necessity of reinforcing manholes and the proper design of heads. A riveted or hammer-welded joint is always much weaker than the plate, however, and if such are used reinforced openings etc. are of no value. To avoid strains in fabrication, steel more than 1 in. thick should be formed hot. The author tests vessels before use with gas oil to a pressure of 75% of the calculated yield point pressure. C. IRWIN.

Treatment of distillates. MORRELL and BERGMAN.—See II. **Syntheses under high pressure.** MORGAN and others.—See III. **Turbidimeter.** HAASE and THIELE.—See XI.

PATENTS.

Heating the charge in rotary furnaces. G. BOJNER and A. P. and A. H. PEHRSON (B.P. 264,825, 12.1.27. Swed., 20.1.26).—The material is heated by a gaseous fluid circulating between the material chamber and an outer chamber rotating with the furnace; in the latter the gas is heated by external means. In some cases the gas may be forced through the material, and if condensable products are given off from the material they may be cooled for the recovery of useful products, the non-condensable part being reheated and recirculated. An intermediate heat-interchanger may be introduced to avoid waste of heat. B. M. VENABLES.

Regenerative furnace. D. B. JONES and F. C. SWARTZ (U.S.P. 1,667,319, 24.4.28. Appl., 28.4.26).—The longitudinal flues below the chequer chamber are directed towards the stack, and each flue has a line of nozzles, built into the floor, pointing towards the stack. Fluid supplied under pressure to the nozzles thus blows foreign matter into the stack. F. G. CLARKE.

Regenerative heat-exchanging device. AKTIEBOLAGET LJUNGSTRÖMS ÅNGTURBIN (B.P. 275,231, 25.7.27. Swed., 31.7.26).—A regenerative mass is provided with longitudinal passages for the two gases (but no transverse passages) and is shaped as a body of revolution, e.g., two segments of a cylinder with two blanked-off smaller segments between. At each end of the active segments partitions (of which the plane contains the axis) oscillate synchronously, and at the ends of the blanked-off portions are chambers forming the inlets and outlets for the two gases; thus, at the end of a stroke, one segment is giving up heat only and the other receiving only, but during a stroke part of each segment is effecting each purpose. B. M. VENABLES.

Mixing machine. H. H. HACKSTEDDE (U.S.P. 1,666,285, 17.4.28. Appl., 21.6.24).—A mixing bowl furnished with heating (or cooling) jacket is arranged to

swing about a vertical hinge at the side. The inlet for heating medium is in the hinge, and the outlet is through a pipe with a swivel on the same axis as the hinge.

B. M. VENABLES.

Rotary drying apparatus. J. B. VERNAY (B.P. 282,432, 16.12.27. Fr., 18.12.26. Addn. to B.P. 262,464; B., 1927, 719).—As the material passes from one concentric cylinder to the next, it is subjected to a grinding action between teeth formed on the rotating end-plates and teeth on the lower part of the fixed casing.

B. M. VENABLES.

Disintegrator. [Emulsifier.] B. CUNIFF, Assr. to COLLOIDAL EQUIPMENT CORP. (U.S.P. 1,666,640, 17.4.28. Appl., 28.4.26).—A pair of discs with intercalating ridges forming a sinuous passage rotate in close proximity at different speeds, the inlet being axial and the outlet for emulsion at the periphery, but one disc is provided with a deep flange which embraces the other disc and causes the final outlet to be comparatively near the axis.

B. M. VENABLES.

Settling tanks. G. S., S. W., and M. J. BINCKLEY (B.P. 287,611, 17.12.26).—A settling tank, for material such as activated sludge in which it is desired that the settled material be discharged promptly, is formed of inverted conical shape, the inlet for sludge being over the whole periphery of the tank, and the outlet for clear effluent through a number of central troughs which are just below the surface of the liquid and are preferably radial. A truncated conical partition is hung within the tank, producing an outer or inlet zone which increases in area downwards. Baffles may be placed in the thick pulp zone to prevent turbulence. B. M. VENABLES.

Centrifugal separators. COMP. IND. DES MOTEURS À EXPLOSION (C.I.M.E.), and D. PERRIER (B.P. 283,963, 19.1.28. Ger., 22.1.27).—The fluid containing solid matter to be separated is passed from an axial inlet to an axial outlet at the other end in a sinuous path through concentric double-conical baffles, so that the material is subjected to a number of treatments with the same (*sic*) centrifugal force.

B. M. VENABLES.

Centrifugal separators. H. LIST (B.P. 285,061, 10.2.28. Ger., 10.2.27).—A centrifugal apparatus is provided with a cover and feed and discharge passages in the hinge thereof, also a valve in the feed which is open when the cover is closed and *vice versa*.

B. M. VENABLES.

Centrifugal separators etc. F. A. G. NOEL and E. W. BAILY (B.P. 288,746, 24.1.27).—The separated liquid collects in an annular chamber formed by an enlargement of the neck of the bowl, and into this annulus of liquid is inserted the annular slit-like mouth of a fixed bell-shaped device, with double walls. The liquid in the space between the walls of the fixed bell rotates more slowly than the liquid outside, producing a difference in pressure which causes the liquid to discharge inwardly and upwardly to a fixed collecting chamber and spout. The result is discharge of the liquid without aeration.

B. M. VENABLES.

Centrifugal. C. SCHAUM, Assr. to FLETCHER WORKS, INC. (U.S.P. 1,667,493, 24.4.28. Appl., 15.1.27).—The driving shaft lies wholly in a stationary tub separated

into two compartments, the lower of which contains turbine blades attached to the shaft and operated by steam, and the upper containing the rotating basket and a governor for regulating the supply of steam to the turbine.

L. A. COLES.

[Rotary] filter. R. A. ELTON (B.P. 287,694, 25.2.27).—The drum of a rotary filter is constructed of two crescent-shaped segments, with longitudinal spaces between so that both the inner and outer surfaces are used for filtering. The filter is operated on the batch system either by pressure with a closed casing or by vacuum with an open casing, and at the start the casing may be completely filled. Detachment of cake is effected by reverse-current flow of liquid or air, and discharge of solid cake by a worm-conveyor in the bottom of the casing, or of sludge, re-formed from the cake and wash liquor, by gravity. Scoops are provided on the edges of the crescents to agitate the pulp and cause a sufficient quantity to flow over the interior filtering surfaces.

B. M. VENABLES.

Rotary drum and vacuum filters. RECKITT & SONS, LTD., and E. H. HALL (B.P. 287,725, 7.4.27).—A rotary drum filter is provided with longitudinal rollers pressing on the cake either directly or through a compression belt, the object being to iron out cracks in the cake. The rollers are given a circumferential movement—preferably reciprocating—over an arc of the filter drum, their position absolute and relative to each other, pressure, and speed being all adjustable.

B. M. VENABLES.

Filters, strainers, etc. V. R. HEFTLER (B.P. 272,236, 7.6.27. U.S., 7.6.26).—A number of washers are assembled on a central bolt with clamping nut; they are stamped in the shape of a wheel with spokes, and alternately with and without a circumferential rim, the latter being thin so that narrow passages will be left for liquid to flow from the spaces between the spokes to the outside, the thickness of the passages being adjustable to a certain extent by compression applied to the pile.

B. M. VENABLES.

Cake former for continuous rotary filters. R. O. BOYKIN, Assr. to N. R. VAIL (U.S.P. 1,666,279, 17.4.28. Appl., 3.3.26).—A pulp distributor is applied to the upper part of a rotary filter drum, comprising a tank of which the bottom is formed by the filtering surface. The distributor is connected to an adjacent pulp reservoir by means which have a certain degree of flexibility.

B. M. VENABLES.

Method of filtering. J. F. WAIT (U.S.P. 1,667,465, 24.4.28. Appl., 10.3.25).—In pressure filtration, a portion of the filter cake formed is removed, the remainder being mixed with the bulk of material within the filter and subjected to further filtration.

H. ROYAL-DAWSON.

Continuous evaporation, distillation, or separation of difficultly distillable liquids. J. Y. JOHNSON. From I. G. FARBERNIND. A.-G. (B.P. 288,775, 17.2.27).—The liquid is sprayed by a disc rotating rapidly about an inclined axis, which itself rotates more slowly about a vertical axis. The spray impinges on the heated walls of a chamber forming a very thin layer, and the

volatile portion evaporates almost instantaneously. Evaporation of easily decomposable substances may be effected at a temperature below the true b.p. by admixture of a volatile fluid (e.g., benzol may be mixed with tar), by the use of vacuum, or by a stream of inert gas.

B. M. VENABLES.

Treatment of gases, vapours, or liquids. CHEM. WERKE CARBON GES.M.B.H. (B.P. 266,673, 29.11.26. Ger., 24.2.26).—Hard, moulded blocks of active carbon manufactured as described in B.P. 247,241 (B., 1926, 308) are used as an absorber. Twenty-one claims are made relating to the properties of the blocks that may be used.

B. M. VENABLES.

Gas and liquid contact apparatus. C. M. ALEXANDER (U.S.P. 1,667,136, 24.4.28. Appl., 27.11.26).—A rectification tower is provided with bubble plates which seal with the casing and in which the down-flows to each storey are at one side in one vertical line, the flow of liquid being across the plate in contact with the gas and back in compact streams to the next down-flow.

B. M. VENABLES.

Separation of liquids or gases. J. HOWARD (B.P. 286,446, 23.2.27).—An apparatus for separating oil from compressed air or similar purposes comprises a curved pipe in which the mixture flows, the oil emerging through a passage in line with the final direction, but the air emerges at right angles to the plane of the curved pipe. Ribs may be formed on the interior of the pipe to increase the surface.

B. M. VENABLES.

Separation of liquids from gases. J. T. H. WARD (B.P. 288,740, 21.1.27).—An apparatus for separating water and/or oil from steam (and similar purposes) comprises a U-shaped conduit in which the fluid is caused to flow in a sinuous motion by baffles which also form pockets to collect the liquid.

B. M. VENABLES.

Apparatus for storing volatile fluids. SULZER FRÈRES SOC. ANON. (B.P. 263,775, 3.12.26. Switz., 30.12.25).—Volatile liquid such as ammonia which is to be delivered partly as vapour and partly as liquid is stored in a container provided with a heating coil and heat insulation; the liquor space is connected to an evaporator to produce the quantity of vapour required, and the vapour space to a compressor and condenser to liquefy any excess vapour formed.

B. M. VENABLES.

Air classifier. A. H. STEBBINS (U.S.P. 1,666,476—7, 17.4.28. Appl., [A] 14.10.26, [B] 16.3.27).—In (A) a spiral conduit is built round a straight vertical pipe, openings being provided in the floor of the spiral and between the spiral and straight pipes. Suction is applied to the top of the straight pipe, and the material is fed to the top of the spiral. As the material runs down the floor of the spiral the finer particles are picked up by the air drawn in through the openings and carried by it to the central pipe. In (B) a long vertical casing formed with circumferential grooves is provided with a co-axial fan or stirrer with blades extending practically the full length of the casing. An upward current of air is produced by another fan, and the material is delivered into the casing at a point intermediate the ends; the lighter particles are drawn out by the air current and the heavier flung by the stirring fan to the sides of

the casing, down which they slide to an outlet at the bottom.

B. M. VENABLES.

Apparatus for separating dust from gases. A. STEVENART (B.P. 284,980, 6.2.28. Belg., 4.2.27).—A cyclone separator is provided with vanes which divide the incoming stream of air and with damping means to collect the dust.

B. M. VENABLES.

Gas separator. J. F. BORDEN, Assr. to OLIVER CONTINUOUS FILTER Co. (U.S.P. 1,667,139, 24.4.28. Appl., 20.3.24).—A vertical, cylindrical casing is provided with a number of horizontal, dished partitions with openings at their centres. Through the openings is inserted a shaft upon which are discs rotating below each opening. Fluid is admitted at the top of the casing and suction applied just below each partition.

B. M. VENABLES.

Identification of crystals. F. F. READ (B.P. 288,496, 19.10.27).—A ray of light is introduced into the crystal and the position of the emerging rays recorded in a suitable way on a target. The crystal is adjusted to a standard position such that the central emergent ray is coincident with the incident ray.

B. M. VENABLES.

Treatment of liquids and gases (U.S.P. 1,668,104).

—See II. **Filter plate** (U.S.P. 1,669,362).—See VII.

Spraying of molten substances (B.P. 276,955—6).—

See X. **Purification of liquids by distillation** (B.P. 287,607).—See XVIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Clean coal in the coking industry. R. LESSING (Gas World, 1928, 88, Coking Sect., 58—62).—In the study of the non-removable constituents of coal, disregard of the difference between ash and mineral matter entails considerable errors. By calculations of the loss of water of hydration in clay and shale, of carbon dioxide liberated from carbonate, and of the addition of oxygen to pyrites and other iron compounds with a coal containing about 10% of ash, the carbon in the pure "ash-free dry" coal may be 3% higher than a corrected value. Similarly, the calorific value and volatile matter are liable to error quite apart from the influence on the latter of the catalytic effect of individual mineral components. To study the constitution of individual mineral components before decomposition and oxidation, consideration must be given to their rôle during coal formation. The composition and content of ash in individual coal components are typical and form a method of identification. The ashes of clarain and vitrain, with a high percentage of water-soluble and low acid-insoluble compounds, represent the original plant ash. The ash of durain, 75% insoluble in hydrochloric acid, is clay substance as proved by its alumina: silica ratio being equal to that of kaolinite. Fusain ash is derived from the carbonates and other salts from water diffused into the decomposing plant remains, and is allied to the segregation of the same substance in the cracks of coal seams in the form of cleat and partings. A water-soluble inorganic accessory of many bituminous coals is sodium chloride. Ash content and composition vary within one group according to particle size: thus, the ash in graded fractions shows great friability of

fusain dust, decrease of ash in clarain with decrease in size and increase in its fines, uniformity in durain and increase of ash in dirt with decrease in size. That coking slack consists of such mixtures of clean coal, dust, and refuse is the basis of all cleaning operations. A coke having 10% of ash is equivalent to 6.7—7% of ash in the coal carbonised. Of this, only 1—1.5% is contributed by the inherent ash, the balance being derived from the dirt. The bulk of the inorganic constituents in coke is inert material which reduces the strength and causes the production of breeze.

H. S. GARLICK.

Influence on the coking of coal of the extraction of the bitumen content with tetralin under pressure.

E. BERL and H. SCHILDWÄCHTER (Brennstoff-Chem., 1928, 9, 121—122).—The bitumen content of ordinary coking coal has been extracted by treating the coal in an autoclave at 250° with tetralin. The extraction of the bitumen has a marked effect in increasing the density of the resulting coke. The solid bitumen compounds have been shown to have a greater effect in this direction than the liquid ones.

A. E. MITCHELL.

Graphitisation of petroleum coke. Relative catalytic effect of various metals. M. L. SCHLEIFSTEIN (Canadian Chem. Met., 1928, 12, 59—61).—When amorphous carbon is heated at a high temperature in the presence of certain inorganic oxides it is converted into graphite. The effect of the addition of different substances on the conversion of petroleum coke has been studied by making rods of the finely-ground coke and 10% of a special pitch; these rods were heated in an electrical resistance furnace at 2400—2500° for 5 hrs. The change was followed by measurement of the resistance of the product, in ohms/cm.³, at a number of temperatures; and it was found that the effect of calcium, manganese, magnesium, aluminium, iron, nickel, chromium, and boron increased in that order. The graphite formed by the use of boron had a resistance only slightly higher than that of Ceylon graphite and below that of Acheson graphite. Treatment of the coke without addition of any catalyst caused partial graphitisation, which may be due to the heat alone or to the presence of inorganic impurities.

R. H. GRIFFITH.

Correlation of physical and chemical properties of cokes with their value in metallurgical processes.

I. W. T. K. BRAUNHOLTZ and H. V. A. BRISCOE (Gas World, 1928, 88, Coking Sect., 63—67).—An attempt is made to correlate certain properties of cokes with their blast-furnace value. Samples have been drawn from the Cumberland and Durham area to conform with certain requirements, viz.:—(a) manufacturing conditions of the coke to be as regular as possible; (b) information to be available as to blast-furnace behaviour; (c) cokes to be of different blast-furnace values; (d) ash content approximately the same. It is found that, in general, there exists a direct relation between the shatter index of a coke and its value in metallurgical processes. In a series of tests to determine the influence of varying the manipulative details of the test as laid down by the U.S. Bureau of Mines, reliable results were obtained from three successive tests, each with 25 lb.

instead of 50 lb. of coke, and investigations into the influence of size of the shatter machine box on the shatter index are being continued in order to standardise the apparatus and eliminate such secondary influences as cushioning of the coke pieces. Repeated shattering of coke remaining on the screen from a former shatter test shows increase in the index; thus, the previous history of a coke sample is of great influence on the observed shatter index. The average weight of pieces of coke of the same size is approximately the same for all cokes examined. In order to study combustibility a special apparatus was designed and information obtained regarding the behaviour of different cokes when burning with a constant and limited supply of air. Gas analyses are conducted after combustion has started, and fuel consumption is calculated from the ratio of carbon dioxide to monoxide. Average sp. gr. and porosities of the cokes examined are tabulated. Investigation of the capacity for water absorption showed that different pieces of coke may absorb different quantities of water during the initial period, but the subsequent steady rate of absorption is practically the same for different pieces of a given coke, and there is no relation between absorption and shatter index or porosity. Preliminary experiments on a coking test of coal in tins indicates that the "box test" is capable of supplying quantitative information as to mechanical strength of coke, and comparison is being extended to other properties.

H. S. GARLICK.

Sardinian fuels, especially those of the Perdasdefogu basin (Ogliastra). II. R. BINAGHI and A. BRUNDO (*Annali Chim. Appl.*, 1928, 18, 122—134; cf. B., 1926, 82).—This district contains anthracite, the quality of which improves as the depth increases. One of the galleries of the Nuraghe S. Pietro mine yielded a sample showing 8.51% of ash and 0.84% of sulphur. The fuel is suitable for producing metallurgical coke, and could be used for smelting the adjacent iron ore, but the nature of the district involves transport difficulties.

T. H. POPE.

Liquid fuels from coal. R. CHAUX (*Bull. Soc. chim.*, 1928, [iv], 43, 385—411).—A lecture.

Rapid determination of the water contents of solid and liquid fuels. J. TAUSZ and H. RUMM (*Gas- u. Wasserfach*, 1928, 71, 417—420).—The fuel is mixed with excess of tetrachloroethane and the mixture distilled through a short column and condenser, the condensate being collected in a burette over mercury. The distillation is continued until the condensate is clear; on being kept the distilled tetrachloroethane and water separate into two layers and the volume of the water is measured. For great accuracy this measurement is made in a capillary burette, arranged to fit by a ground joint to the usual collecting burette, into which the liquid is forced by the mercury column. The method has been tested against the practice of drying the materials under a vacuum at various temperatures and, in general, the agreement by the two methods is satisfactory. Application of the method to the determination of moisture in material from gas purifiers gave more divergent results. For the method it is claimed that a complete determination

can be made in 20 min., that owing to the large quantities of material that can be employed and the excellent separation of water from tetrachloroethane accurate results are possible, that owing to the non-inflammability of the tetrachloroethane the risks of fire attendant on the xylol method are obviated, and that the total water content, free and combined, of the material is given.

A. E. MITCHELL.

Low-temperature carbonisation. F. SCHWERS (*Chim. et Ind.*, 1928, 19, 583—588).—The Salerni carbonisation process, which is suitable for treatment of shales, lignites, etc., can be used for bituminous coal, although further experience will be necessary before such applications are perfectly successful. Work in this direction is proceeding at Ghent; at Zagreb a local deposit of lignite will be carbonised; at Sarrebruck dust coals of various types, and at Resiutta a bituminous shale are treated. No details as to the yield and nature of the tars are yet available.

R. H. GRIFFITH.

Apparatus used in the distillation of coal, shale, and petroleum products by official methods. F. BORDAS (*Ann. Falsif.*, 1928, 21, 146—154).—The standard distillation apparatus used in France for the grading of petroleum and coal-tar oils is described, and reference is made to the methods employed in other countries.

T. M. A. TUDHOPE.

Removal of hydrogen sulphide from industrial gases by alkaline potassium ferricyanide solutions. F. FISCHER and P. DILTNEY (*Brennstoff-Chem.*, 1928, 9, 122—126).—The factors governing the removal of hydrogen sulphide from industrial gases by alkaline potassium ferricyanide solutions according to the reaction $2K_3Fe(CN)_6 + H_2S + 2KOH = 2K_4Fe(CN)_6 + S + 2H_2O$ have been investigated. A loss of sulphur, the extent of which depends on the alkalinity of the solution, occurs through secondary reactions. This loss was reduced to a minimum by employing solutions containing 1 mol. of sodium carbonate and 2 mols. of sodium bicarbonate to each mol. of the ferricyanide. The difficulty of regenerating the ferrocyanide liquors, encountered by Glud and Schönfelder (B., 1927, 321), has been overcome by using electrolytic methods based on the results of von Hayek (B., 1904, 493) and others. The results form the basis of a regenerative process for the removal of hydrogen sulphide from industrial gases with a maximum recovery of sulphur.

A. E. MITCHELL.

Removal of carbon dioxide from industrial gases with alkali carbonates at normal pressures. F. FISCHER and P. DILTNEY (*Brennstoff-Chem.*, 1928, 9, 138—144; cf. B., 1926, 906).—Several examples of absorption of carbon dioxide by solutions of alkali carbonates are described in the literature, but these are mainly concerned with the preparation of the gas in an air-free condition. A gas containing 21.8% CO_2 was washed with 5% potassium carbonate solution at 23°, and 2 litres of the liquid were found sufficient for 60 litres of gas; the amount of water needed to produce the same effect would be very much greater. The washing becomes less efficient as bicarbonate accumulates in the liquid, but the solution can be

regenerated by boiling or by blowing air through it when warm; passing a stream of air through the solution under reduced pressure is not satisfactory even at 60°. Experiments with granules of solid sodium hydroxide in the presence of limited amounts of water vapour were not successful as the reaction between carbon dioxide and the solid was too slow; it was found that sodium carbonate absorbs less carbon dioxide in a given time than the potassium salt, but could be more rapidly regenerated. Absorption was assisted by warming the solution to about 70–80°, and regeneration proceeded best at 90–95°. A tower for the continuous treatment of gas in this way is 80 cm. high and 8 cm. in diam., and stands immediately above a similar tower, and liquor from the first, which has absorbed carbon dioxide, is revived by an air-blast in the second; regenerated solution is pumped back to treat more gas. The success of the process depends on adjustment of the relative rates of flow of gas, air, and liquor, on the temperature and concentration of the solution, and on the nature of the packing in the tower.

R. H. GRIFFITH.

Determination of oxygen in illuminating gas. V. FUNK (Gas- u. Wasserfach, 1928, 71, 443–444).—Analysis of gases containing small amounts of air cannot be accurately carried out by the Lubberger method, and an improved form of absorption vessel has been designed. This is used on the Orsat principle, and can be filled with either pyrogallol solution or with a paste of chromous acetate; the latter has the advantage that preliminary removal of carbon dioxide is not necessary. In order to obtain accurate results it is essential to saturate the liquids with gas before use, and to prevent wide fluctuations in the temperature of the apparatus. Under these conditions oxygen can be determined to within 0.01%.

R. H. GRIFFITH.

Synthesis of higher hydrocarbons from water-gas [at atmospheric pressure]. D. F. SMITH, J. D. DAVIS, and D. A. REYNOLDS (Ind. Eng. Chem., 1928, 20, 462–464).—Pure dried water-gas was passed over a heated catalyst and the exit gases were cooled with liquid air. Of various catalysts tried “promoted” iron and cobalt chromate were found inactive, but several mixtures of cobalt oxide with other metallic oxides gave small proportions of liquid hydrocarbons. The oil yield was a maximum at 275°. The products also included carbon dioxide, water vapour, and methane. The highest yield of liquid hydrocarbons found was 66 g./m.³ of gas. From 2 to 3% of the carbon monoxide used appeared as carbon deposited on the catalyst. A very low rate of gas flow was also essential.

C. IRWIN.

Determination of free carbon in tars, pitches, etc. E. BERL and H. SCHILDWÄCHTER (Brennstoff-Chem., 1928, 9, 137–138).—Extraction of tars with solvents such as benzene, toluene, etc. leaves a residue which consists of a mixture of true “free carbon” and bituminous substances. It is possible to separate the latter only by passing the tar through a suitable sintered glass filter, but this method is laborious, and the use of tetralin under pressure at 250° to extract soluble material is found to give satisfactory concordant results.

R. H. GRIFFITH.

Phenol recovery plant avoids waste pollution of streams. H. E. JONES (Chem. Met. Eng., 1928, 35, 215–218).—Trouble has arisen through the discharge of ammonia-still effluents containing phenol into rivers the water from which is used for public supply after chlorination, the taste of chlorophenol even in minute proportions being very marked. In such circumstances the recovery of phenol from crude ammonia liquor by extraction with benzol has been developed. Steel towers in pairs are used with perforated plate and coke distributors and countercurrent feeds in series. Two discontinuous caustic soda washers are used alternately, the sodium phenoxide being decomposed with sulphuric acid. 1800 gals./hr. of benzol are used against 1500 gals. of ammonia liquor. The benzol recovery and the extraction efficiency are each 96%. Benzol losses include a small proportion dissolved in the ammonia liquor and eventually returned to the gas stream and are otherwise minimised by the provision of seals to reduce the effect of the alternate inflow and outflow of air in the system. The part-time attendance of one operator is sufficient.

C. IRWIN.

Recovery methods and the ammonia problem. L. H. SENSICLE (Gas J., 1928, 182, 384–390).—A historical review of the development of ammonia recovery and a description of the semi-direct and Otto and Simon-Carves direct recovery processes. An efficient type of saturator and results obtained therewith are described. The possibility of cheapening recovery of ammonium sulphate at gas-works by the adoption of direct-recovery methods is discussed, and an attempt made to determine the conditions necessary for successful operation. Attempts made to eliminate the cost of the sulphuric acid by utilising the sulphur in the gas, which is in excess of that required to provide the sulphate radical required by the ammonia, have met with limited success. Calcium sulphate is used extensively for synthetic sulphate manufacture, but is only suitable for very large ammonia output plant. In a process recently developed by the I. G. Farbenindustrie A.-G. (cf. B.P. 285,999; B., 1928, 325), the ammonia is fixed as thiosulphate and polythionate, the success of the process depending on the market outlet for the mixed salts produced. The direct process of ammonia recovery is shown to have potentialities for the successful solution of the effluents problem since by this process they would be reduced to about one fifth of the amount usually present. The noxious constituents are practically confined to phenolic substances which exist in sufficient concentration to render probable their successful extraction by solvents such as benzol. Further attempts to solve the effluent problem without adding to the present cost of sulphate recovery and to produce sulphate with a saving of probably 5% of the selling price are being undertaken.

H. S. GARLICK.

Neutralisation of naphtha-yielding oils and a new dry-refining process. E. T. LEEMANS (Petroleum, 1928, 24, 548–549).—A mid-American oil could not be refined by the usual methods owing to the presence of free naphthenic acids in the crude. The latter was therefore heated to 40–100° in a preheater, neutralised by agitation for 1 hr. with the calculated quantity ($\pm 0.5\%$) of dry, powdered, slaked lime, and

allowed to settle prior to distillation. This was preferably carried out under a high vacuum and with a plentiful supply of steam, when the calcium salts of the naphthenic acids remained in the residual asphalt, the properties of which were not appreciably affected thereby. The asphalt could not, however, be used in the preparation of asphalt emulsions. The lubricating oil fractions, after acid treatment and before neutralisation, were found to be strongly acid, owing to the presence of sulphonic acids in the acid tar dissolved in the oil. The latter (6000 litres) was agitated with a solution (40 litres) of water-glass (9 litres of water; 1 litre of water-glass [d 1.38]), whereby sodium sulphonates and free silicic acid were formed, the latter causing the coagulation of the acid tar particles and, by adsorption, preventing dissolution of the sulphonates in the oil. Precipitation was completed in a flat settling vessel at 40–50°. The oil was then pumped to a bleacher, heated at 100°, and neutralised by agitation with powdered slaked lime (1–2%). A filter-aid was added and the oil filtered through a press. The refined product was orange-yellow to yellow, and showed no tendency to emulsify with water. It was of good odour, and its setting point was lowered from +2° to –6°, probably due to adsorption of impurities by the silicic acid gel formed.

R. C. ODAMS.

Thermal decomposition of organic sulphur compounds. W. F. FARAGHER, J. C. MORRELL, and S. COMAY (Ind. Eng. Chem., 1928, 20, 527–532).—The following organic sulphur compounds, being selected as representative of those types which are considered to be present in petroleum, were dissolved in naphtha made from Pennsylvania crude oil, and the vaporised solutions subjected to an average temperature of 496°: mercaptans, alkyl sulphides, alkyl disulphides, thiophen, and elementary sulphur. The products of the thermal decomposition have been identified. Comparative tests have been made with the products of cracking of solutions of *n*-butyl and isoamyl disulphides in gas oil at 437° and 17 atm. The types of sulphur compounds produced were the same as those produced at atmospheric pressure for both disulphides. A suggested mechanism for the thermal decomposition is outlined. E. H. SHARPLES.

Ability of insulating oils to resist oxidising influences. S. FACHINI and C. BORELLA (Chem. Umschau, 1927, 34, 331–334).—Under the influence of atmospheric air and high temperature a gradual change in mineral oils occurs which is indicated by the darkening of the oils, by the formation of asphaltenes insoluble in light petroleum, by the formation of such acidic products as asphaltogenic acids and fatty acids of varying solubility according to the nature of the solvent, and by the formation of such fugitive substances as lower organic acids, water, and carbon dioxide. The formation of such oxidation products is dependent on catalytic influences, on the action of metals, the presence of fatty acids, the chemical nature of the oils themselves and of the extent to which they have been refined, and the influence of electrostatic fields. The various English, American, and Continental methods for testing oils, with a view of judging their ability to resist oxidation, are described. Details are also given of the various tests

applied to determine the amounts of oxidation products, saponifiable material, and acid contents of the oils, the methods including gravimetric, volumetric, and solubility determinations. The destructive action of the oils on the insulating material is also determined.

H. M. LANGTON.

Changes in transformer oils. II. K. TYPKE (Z. angew. Chem., 1928, 41, 418–425).—A review of recent literature dealing with the nature of the changes which take place in transformer oils during use, the products formed, and precautions to be taken to retard the progress of these changes.

A. R. POWELL.

Detection and determination of light petroleum, benzene, alcohol, ether, and tetralin in motor fuels.

FORMÁNEK (Chem.-Ztg., 1928, 52, 325–326, 346–348).—The vat dyes Algol Red BTK and 2G colour benzene, its homologues, and tetralin red, and Lake-Red Ciba B colours them rose-red, whereas light petroleum, gasoline, alcohol, and ether remain practically colourless, so that a close approximation of the benzene content of a motor fuel may be obtained by shaking 20 c.c. of the fuel with 0.02–0.05 g. of the dye for 2 hrs. and comparing the colour with standards produced from mixtures containing known amounts of benzene. Aniline Blue 2B colours alcohols, aldehydes, and ketones a deep blue, but is completely insoluble in water, benzene, ether, or petroleum products. Ether is detected by the blue perchromic acid test. A liquid fuel containing less than 20% of tetralin, benzene, ether, or alcohol may be tested for light petroleum by warming with aniline, which dissolves in the warm liquid, but separates on cooling. Alcohol is determined by shaking 100 c.c. of the fuel with 150 c.c. of water coloured with fuchsin, and measuring the colourless layer which represents the constituents other than alcohol. To determine ether in an alcohol-ether mixture, 100 c.c. of the liquid are mixed with 50 c.c. of light petroleum, and the mixture is shaken with 100 c.c. of coloured water. The volume of the top layer, less 50 c.c., is the volume of ether in the mixture. Tetralin is determined colorimetrically by means of Algol Red BTK in the residue left after distillation of the mixed fuel at 200°.

A. R. POWELL.

Designing equipment for chemical treatment of oil distillates. J. C. MORRELL and D. J. BERGMAN (Chem. Met. Eng., 1928, 35, 210–214).—The orifice column is the most satisfactory device for the continuous mixing of oil with acid or other reagents. Several small holes with a fairly high pressure drop at each orifice plate should be used. The flow in cub. ft./sec. is $0.65A\sqrt{2gh}$, where A is the orifice area and h the head, both in feet. Several methods of construction are described, and experimental data for the pressure drop in columns packed with 1 in. Raschig rings and screened gravel are given. The friction rises more rapidly than the rate of flow. Jet mixers are efficient, but the time of contact is short, and they are not suited to the problem of mixing a large volume of oil with a small volume of reagent. A centrifugal pump with the impeller cut down and running in the reversed direction makes a good mechanical mixer. A baffle-plate column is not an efficient mixer in itself, but is a useful auxiliary to an orifice plate column.

C. IRWIN.

British coking industry and some of its products.

R. RAY (J. Inst. Fuel, 1928, 1, 220—233).

Utilisation of town's refuse and refuse fuels.

A. B. SCORER (J. Inst. Fuel, 1928, 1, 288—300).

Chemical study of processes involving carbonisation of coal by internal heating. M. W. TRAVERS (J. Inst. Fuel, 1928, 1, 272—278).—See B., 1928, 77.

Vertical intermittent chamber ovens for gas manufacture. N. J. BOWATER (J. Inst. Fuel, 1928, 1, 234—245).—See B., 1928, 76.

Liquid fuels other than petroleum. A. E. DUNSTAN and H. G. SHATWELL (J. Inst. Fuel, 1928, 1, 262—271).—See B., 1928, 290.

Viscosity of oils. RAASCHOU.—See I. **Hydrocarbons from fats.** MELIS.—See XII. **Butadiene.** BIRCH.—See XIV.

PATENTS.**Machines for separating coal and other materials.**

W. H. BARKER (B.P. 288,786, 1.3.27).—Coal is caused to slide down an inclined plate, the surface of which is made of glass. The coal acquires a higher velocity than the impurities, and therefore can jump a larger gap at the end of the plate. Suitably-placed chutes receive the coal, impurities, and intermediate substances. The end of the inclined plate is made of polished stainless steel or material less resilient than glass, which prevents bouncing of the coal. T. A. SMITH.

[Smokeless] coal-ball manufacture. C. B. WISNER (B.P. 275,602, 25.7.27. U.S., 7.8.26).—Ground bituminous coal is subjected to a preliminary heat-treatment in the presence of air before it is carbonised and converted into balls by passage through an externally-heated rotary retort. During the pretreatment a temperature is maintained just insufficient to distil the hydrocarbons, and by regulating the time of treatment and the quantity and temperature of the air the amount of binder present is controlled, whereby sticking of the charge to the walls of the retort during carbonisation is prevented, and the strength and average size of the product are determined. Complete removal of hydrocarbons is not effected and cracking is avoided. The temperature employed is below that at which the coal ceases to expand, in order to prevent fracture of the product. A strong coking coal, asphalt pitch, or pitch from the oil obtained in the process itself is added when non-coking coal is used, either during the pretreatment or during the carbonisation. Suitable apparatus is described. F. G. CLARKE.

Manufacture of briquettes from low-volatile fuels, fine ores, flue dust, etc. with the aid of binders. KOKS- U. HALBKOKS-BRIKETTIERUNGS-GES. M.B.H. (B.P. 274,876, 20.7.27. Ger., 24.7.26).—By adding sulphite waste liquor or cellulose pitch to the clay binder used in the manufacture of briquettes, the mechanical strength of the latter is considerably increased, without appreciable increase in the ash content, and the good qualities which characterise clay briquettes are retained. C. O. HARVEY.

Manufacture of fuel briquettes. J. S. ROBESON (U.S.P. 1,667,358, 24.4.28. Appl., 26.9.24).—A tubular

briquette formed from fine coal, oil, and lignone, the outer portion being harder than the inner, and containing a lower percentage of volatile matter.

H. ROYAL-DAWSON.

Treatment of fine coal. E. W. WILKINSON, ASST. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,667,277, 24.4.28. Appl., 4.8.26).—The fine coal is agitated with water and less than 1% of an organic compound containing sulphur, which assists agglomeration under such conditions that the coal becomes coated with oil and floats. It is separated and freed from excess water. F. G. CLARKE.

Distillation of fine granular coal or bituminous material. TROCKNUNGS-, VERSCHWELUNGS-, U. VERGASUNGS-GES.M.B.H., L. HONIGMANN, and F. BARTLING (B.P. 289,137, 18.1.27).—Contrary to previous experience with the dry distillation of fine granular coal, peat, etc. by a continuous process in which the material is spread in a layer on a revolving annular horizontal hearth in a furnace, it has been found that, owing to the rapid and complete heating attainable, the throughput may be greatly accelerated, and the output considerably increased by employing shallow layers of material of 1—3 mm. thickness. C. O. HARVEY.

[Batch] distilling apparatus for carbonaceous fuel. Carbonising process. W. E. TRENT, ASST. to TRENT PROCESS CORP. (U.S.P. 1,668,131—2, 1.5.28. Appl., [A] 14.10.22, [B] 11.3.25).—(A) A vertical, cylindrical distillation chamber contains upon its base an electric grid having upward projections. (B) Charges of fuel introduced into a chamber by means of an endless conveyor are heated therein by a superposed heat-storage element. Gases are removed from the chamber from a point below the charge. F. G. CLARKE.

Retorts for the distillation of lignite and other carbonaceous materials. R. W. EASTON (B.P. 288,700, 11.1.27).—A vertical retort may be tapered and is divided by vertical partitions; both may be of cast iron. The water vapour and poor gases are exhausted at the top, whereas the rich gas is exhausted through horizontal conduits crossing the retort at some point intermediate between the top and bottom. These conduits are of circular form, with openings only on the underside or of inverted U-shape. B. M. VENABLES.

Production of power [from low-grade fuel]. M. J. TRUMBLE (U.S.P. 1,667,403, 24.4.28. Appl., 25.10.22).—Steam is blown through a retort containing low-grade fuel, and the hydrocarbon vapours liberated are passed with the steam to a power unit such as a turbo-generator. The condensable hydrocarbons are recovered from the condenser of the turbine.

T. A. SMITH.

Manufacture of fuel gas. I. G. FARBENIND. A.-G. (B.P. 269,594, 19.4.27. Ger., 19.4.26).—Granular fuel is gasified in a horizontal tube lined with refractory material, along which the glowing fuel is carried with a hopping or jumping motion by a current of the gasifying agent; or the fuel may be partially gasified in the above manner and then passed into a separator which is operated as an ordinary gas producer.

A. B. MANNING.

Manufacture of water-gas. N. J. BOWATER (B.P. 288,797, 17.3.27).—A downward stream of pulverised fuel reacts with an upward stream of steam introduced at the bottom of a reaction chamber, which contains a heat-radiating surface. The preferred apparatus consists of a vertical chamber, the circular cross-section of which is greater at the top than at the bottom, in order to reduce the speed of the rising gases and thus prevent unburned carbon being carried away. The chamber should be lined and lagged. The heat-radiating surfaces may consist of an earthenware cylinder heated to incandescence by the combustion of gaseous, liquid, or solid fuel in its interior. The products of combustion may be used to preheat the air and fuel used for the combustion or for raising steam. The heat for the reaction may also be supplied by any hot gases or electrically.

F. G. CLARKE.

Generation of water-gas from bituminous fuel. O. MISCH (B.P. 288,426, 21.3.27).—A water-gas generator is surmounted by a retort in such a way that the distilled fuel falls by gravity into the generator (cf. B.P. 276,530; B., 1927, 867). The retort is heated externally at its lower end by water-gas, using a specially designed compressed-air burner, by oil or by powdered fuel, in such a way that the temperature is higher than 700° at the lower end of the retort, and falls to about 250° at the charging end. Oil or tar may be injected into the incandescent coke layer in the retort in order to raise the calorific value of the gas. A short intermediate period may be arranged between the "blow" and the "run" in the producer, during which a little steam is passed for scavenging the remainder of the nitrogen and carbon dioxide.

A. B. MANNING.

Manufacture of carburetted water-gas. F. B. HAYES, ASSR. to WESTERN GAS CONSTRUCTION CO. (U.S.P. 1,667,518, 24.4.28. Appl., 25.9.24).—Up-run water-gas is passed through a carburettor where it is enriched by admitting oil. The down-run blue water-gas is by-passed with respect to the carburettor, which during this part of the cycle is employed to make oil-gas. The blue water-gas, carburetted water-gas, and oil-gas are finally mixed in a holder.

T. A. SMITH.

Gas producers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 288,491, 8.10.27).—Finely powdered or granular fuel is gasified in a disc-shaped chamber set with its axis horizontally. The fuel and the gasifying media (air and steam) are admitted tangentially, through a number of openings or jets in the periphery, in such a way that the fuel particles and gases rotate with a high velocity and the fuel is rapidly gasified. The gaseous products and ash are led away through a central opening in one side of the chamber. The combustion and reduction zones may be separated by an annular wall within the chamber. The producer may be used as a burner, the air for combustion being supplied to the gas at its point of exit from the chamber. A horizontal extension pipe, preferably with openings for the admission of additional gases, may be attached to the discharge opening of the chamber.

A. B. MANNING.

Water-gas plant. POWER-GAS CORP., LTD., and N. E. RAMBUSH (B.P. 288,463, 6.7.27).—The plant

includes a steam superheater and waste-heat boiler, the superheater being of the U-tube type and having its tubes covered with non-metallic protective material of high heat-storage capacity; this material serves also as an igniter for the mixed "blow" gases and air. The water space near the top of the boiler tubes is made of larger diameter than the rest of the boiler, and a plurality of pipes is provided between the steam drum and the top of the boiler in order to facilitate the removal of steam.

A. B. MANNING.

Manufacture of hydrogen or gas mixtures containing hydrogen from hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND A.-G. (B.P. 288,662, 15.11.26; cf. G.P. 403,049; B., 1925, 71).—Hydrocarbons are partially burned by means of oxygen or air enriched with oxygen, possibly with the addition of water vapour, and the resulting gases treated with activated catalysts which facilitate the conversion of a mixture of hydrocarbons, carbon monoxide, and steam into a mixture containing hydrogen and carbon monoxide or dioxide. When carbon monoxide is not desired in the product, steam is added during the second stage and suitable catalysts are used to give a mixture of hydrogen and carbon dioxide, the latter being finally removed, if desired. More than one catalyst may be employed; e.g., the preheated gases may first meet a heat-resisting catalyst and then a more active catalyst. The partial combustion may be effected by means of flame or by surface combustion, with employment of solid materials upon which catalysts may be deposited. The temperature of the partial combustion is reduced by freeing the hydrocarbons from organic and inorganic sulphur compounds. Instead of subjecting the whole of the gases to partial combustion, a portion may be burned and the products mixed with the remaining hydrocarbons. Thermal decomposition may be employed in the first stage of the process instead of partial combustion. The hydrocarbons may be passed through externally heated tubes and the resulting carbon collected; or, in a discontinuous process, the gases are passed in contact with hot fireproof materials, which are reheated at a subsequent stage by burning the deposited carbon. The decomposition may be facilitated by adding carbon dioxide, oxygen, or steam.

F. G. CLARKE.

Washing or cleaning of gas. H. A. BRASSERT & Co. From H. A. BRASSERT (B.P. 289,118, 24.12.26).—A gas washer suitable, for instance, for blast-furnace gases, and of robust and inexpensive construction, consists of a tower filled with bundles of wooden hurdles and provided with water sprays. The hurdles are of graded mesh, those at the base of the tower having the largest apertures. The arrangement is such that the gases follow a tortuous path, and are given a preliminary treatment involving alternate compression and expansion.

C. O. HARVEY.

Bituminous emulsions. J. A. MONTGOMERIE (B.P. 288,821, 23.4.27).—The process of B.P. 226,032 (B., 1925, 91) is modified by the use of solutions of alkali carbonates or bicarbonates in place of the hydroxides, whereby precipitation of calcium and magnesium salts, present in the water used, is effected, and breaking of

the emulsion due to the formation of insoluble organic compounds of these metals is avoided.

C. O. HARVEY.

Coating of material [paper] with bituminous substances. N. V. DE BATAAFSCHE PETROLEUM MAATSCHAPPIJ (B.P. 273,281, 16.6.27. Holl., 28.6.26).—The paper pulp is mixed with asphalt in a "gel" form, obtained by treating an emulsion of asphalt with an electrolyte in presence of a protective colloid such as albumin.

C. O. HARVEY.

Recovery of ammonia from ammoniacal liquor. SOUTH METROPOLITAN GAS CO., P. PARRISH, F. C. SNELLING, and O. W. WEIGHT (B.P. 288,436, 13.4.27).—Waste gases from the retort settings pass through a waste-heat boiler and then through a preheater. The ammoniacal liquor is passed through the preheater on its way to the ammonia still, which is operated by steam from the waste-heat boiler.

A. B. MANNING.

Manufacture of motor fuels. I. G. FARBERIND. A.-G. (B.P. 278,341, 2.10.26. Ger., 5.11.25).—The fuels consist of mixtures of natural benzine, aliphatic alcohols (e.g., methyl alcohol), and products obtained by destructive hydrogenation. Such products, unlike pure natural benzines, are readily miscible with the lower alcohols (cf. B.P. 259,944; B., 1928, 222).

C. O. HARVEY.

Motor fuel. T. MIDGLEY, JUN., ASSR. to GEN. MOTORS CORP. (U.S.P. 1,668,022, 1.5.28. Appl., 6.1.25).—The fuel contains a hydrocarbon, a volatile compound of a metal, and a halogenated lubricating oil.

H. ROYAL-DAWSON.

Manufacture of synthetic liquid fuels. SOC. INTERNAT. DES PROC. PRUDHOMME HOUDRY (B.P. 275,585, 12.7.27. Fr., 3.8.26).—Petroleum and similar fuels are produced by hydrogenation of the vapours obtained by distillation of materials such as lignite, peat, shales, tars, heavy oils, etc. in a chamber containing no catalyst. The hydrogen is rendered active by treatment with a catalyst in a separate chamber, fouling of the catalyst being thus avoided (cf. B.P. 274,846).

C. O. HARVEY.

Gasification of liquid fuels. GASIFIER CO., ASSEES. of G. L. REICHHHELM (B.P. 277,604, 4.1.27. U.S., 16.9.26).—Liquid fuel is gasified in a specially designed apparatus by injecting air and fuel into an enclosed space and supplying air to promote an enshrouding zone of complete combustion (indicated by a blue flame). A catalysing medium situated in this zone effects gasification of the fuel.

C. O. HARVEY.

Manufacture of lubricating oils. F. W. HALL, ASSR. to TEXAS CO. (U.S.P. 1,668,313, 1.5.28. Appl., 5.10.23).—Sulphuric acid is added to cylinder oil, and the treated oil mixed with a lighter petroleum oil and allowed to settle from the resulting acid sludge.

H. ROYAL-DAWSON.

Decolorisation of [lubricating] oils at high temperatures. P. W. PRUTZMAN, ASSR. to CONTACT FILTRATION CO. (U.S.P. 1,667,984, 1.5.28. Appl., 6.12.26).—A stream of oil containing an absorbent is heated to a temperature above that of vaporisation of the oil, and is subsequently cooled below that temperature, so that no vapours are lost.

C. O. HARVEY.

Manufacture and treatment of lubricating grease for use with grease guns etc. C. BREWER (B.P. 288,796, 17. and 22.3.27 and 13.5.27).—A lubricating grease which can be moulded into bars and remains sufficiently solid to be handled under any climatic conditions is made by mixing a lubricating distillate from a paraffin or other suitable base-oil with stearine or a stearine compound (tallow, soap, etc.), distilling the mixture with superheated steam and a current of oxygen, and re-combining distillate and residue after freeing the latter from asphalt.

C. O. HARVEY.

Treatment of hydrocarbon oils. W. M. CROSS, ASSR. to GASOLINE PRODUCTS CO. (U.S.P. 1,667,883, 1.5.28. Appl., 20.7.22. Renewed 5.3.28).—The oil is fractionally distilled and the fractions are subjected to processes of refining and redistillation. Portions of the residues and higher fractions are cracked under pressure, and the resulting cracked liquids are recirculated through the fractional distillation plant.

C. O. HARVEY.

Removal of sulphur and its compounds from hydrocarbon oil. G. L. WENDT, ASSR. to STANDARD OIL CO. (U.S.P. 1,668,225, 1.5.28. Appl., 16.7.24).—Lower mercaptans are added to the oil, which is then treated with "doctor solution."

H. ROYAL-DAWSON.

Treatment of hydrocarbons. W. HESS (U.S.P. 1,668,275, 1.5.28. Appl., 7.9.26. Ger., 25.9.25).—The hydrocarbons are pretreated with gaseous and then with liquid sulphur dioxide.

H. ROYAL-DAWSON.

Oxidation of mineral hydrocarbons. J. H. JAMES (U.S.P. 1,667,419, 24.4.28. Appl., 6.12.21).—A mixture of the vaporised hydrocarbons and a gas containing oxygen is brought into contact with a catalyst at a temperature sufficiently high to bring about partial oxidation.

C. O. HARVEY.

Refining of petroleum products. F. B. THOLE, S. F. BIRCH, and W. S. G. P. NORRIS (B.P. 288,931, 13.10.26).—Petroleum fractions boiling within the benzene-kerosine range are decolorised and deodorised by a refining process, the novel step in which is a treatment with a solution of an alkali hypochlorite containing not less than 1.5% of free caustic alkali. The objectionable sulphur compounds are thus rendered easily removable by subsequent acid-alkali refining and distillation. The fractions may also undergo partial refining prior to the hypochlorite treatment.

C. O. HARVEY.

Apparatus for treating and combining hydrocarbon liquids and gases and other liquids and gases. N. H. COLWELL (U.S.P. 1,668,104, 1.5.28. Appl., 29.4.24).—The apparatus consists of a chamber into which liquids may be pumped under pressure through vaporising nozzles.

C. O. HARVEY.

Removal of wax from oil. G. D. GRAVES, ASSR. to STANDARD OIL CO. (U.S.P. 1,668,239, 1.5.28. Appl., 27.7.25).—The waxes which hinder filtration or sweating of ordinary wax from oil distillates are removed prior to the final de-waxing process by filtration at a temperature at which the ordinary wax remains in solution, but below that temperature at which crystallisation of the objectionable waxes occurs.

C. O. HARVEY.

Fractionation of oils. C. M. ALEXANDER (U.S.P. 1,667,340, 24.4.28. Appl., 12.1.26).—The vapours obtained by distillation of the oil are brought into contact with reflux condensate of which the final b.p. is lower than that of the highest-boiling constituent of the vapours. The vapours are then brought into contact with a higher-boiling condensate.

C. O. HARVEY.

Distillation [of various substances] and generation of [high-pressure] steam. T. CHMURA (B.P. 259,214, 29.9.26. Ger., 29.9.25).—Benzene vapour, which has a heat capacity at 350° of about $4\frac{1}{2}$ times that of superheated steam and a decomposition temperature of about 800°, is used as a circulating heat-carrier in the distillation of the high-boiling fractions of petroleum, lignite, oil shales, etc., or for heating a boiler for extra-high-pressure steam. The benzene may give up its heat through a separating wall or directly to the material. For the latter case, methods, *e.g.*, rectification, are described for purifying and avoiding loss of benzene. To avoid decomposition of benzene, the heater and conduits may be lined with tin or ferrous sulphide, or a proportion of hydrogen may be introduced. Low-value heat carriers such as steam, carbon dioxide, or natural gas may also be admixed to a moderate extent.

B. M. VENABLES.

Manufacture of fuel briquettes. A. M. HART, Assr. to HART CARBON FUEL CO., LTD. (U.S.P. 1,668,643, 8.5.28. Appl., 9.8.27. U.K., 22.4.26).—See B.P. 279,140; B., 1928, 79.

Treatment of pulverised vegetable fuels. S. SHIMAMOTO (U.S.P. 1,668,660, 8.5.28. Appl., 1.11.26).—See B.P. 273,556; B., 1927, 693.

Carbonisation of coal. W. RUNGE, Assr. to INTERNAT. COAL CARBONIZATION CO. (U.S.P. 1,669,023—4, 8.5.28. Appl., 6.11.24).—See B.P. 242,622; B., 1926, 428.

Composition to be used in connexion with anti-knock compounds. W. GAUS, Assr. to I. G. FARBE-ENIND. A.-G. (U.S.P. 1,666,693, 17.4.28. Appl., 17.4.25).—See B.P. 252,019; B., 1927, 357.

Treatment of emulsions [of hydrocarbon oils and water]. C. F. PESTER, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,669,379, 8.5.28. Appl., 20.3.23).—See Can. P. 247,810; B., 1927, 403.

Evaporation etc. of liquids (B.P. 288,775). Treatment of gases etc. (B.P. 266,673)—See I. **Lead sludge** (U.S.P. 1,667,550). **Production of gas** (U.S.P. 1,667,272).—See VII. **Cement from oil shale** (U.S.P. 1,665,993).—See IX.

III.—ORGANIC INTERMEDIATES.

Syntheses under high pressure. Interaction of carbon monoxide and hydrogen. G. T. MORGAN, R. TAYLOR, and T. J. HEDLEY (J.S.C.I., 1928, 47, 117—122 τ).—A study has been made of the interactions occurring between carbon monoxide and hydrogen. The preparatory experiments were made in a static apparatus consisting of a hydraulic compressor through which the mixed gases were forced at 200 atm. into small copper-lined steel reaction vessels charged with

the catalytic mass and heated in a metal bath to 280—410°. The relative efficacy of each catalyst was ascertained by noting the time required for a drop in pressure of from 10 to 20 atm. These catalysts had zinc oxide as a foundation, and this basic oxide was employed either alone or in combination with the acidic oxides of chromium, manganese, molybdenum, tungsten, uranium, and vanadium. For special purposes other basic oxides such as those of cobalt and copper were added to the zinc oxide. These tests indicated that a catalytic mass containing zinc and chromium oxides would probably prove to be an efficient catalyst when employed at 350—400° and 200 atm. Flow-through experiments were then made in apparatus consisting of a Reavell three-stage compressor, a Lennox circulatory pump, a set of three copper-lined steel reaction vessels heated in a bath of molten lead, and an efficient cooling and condensing plant. Each vessel was fitted with inlet and outlet tubes, each of the latter communicating with its own condenser and receiver. Since the circulating gases retained an appreciable amount of the more volatile products of reaction, these gases, after passing from the condensers, circulated through tubes cooled in brine by a sulphur dioxide "frigidaire" refrigerator which operated automatically in reducing the temperature. In this flow-through plant condensations were effected both with a basic zinc chromate catalyst and with admixtures of this material with cobalt chromate. With the latter catalyst, in addition to the main product, methyl alcohol, there was obtained a notable proportion of ethyl alcohol together with such higher primary alcohols as propyl and isobutyl alcohols. Moreover, the organic distillates contained acetaldehyde, propaldehyde, butaldehyde, and other higher aldehydes. The chemical changes involved may be regarded as arising from aldolisation of formaldehyde which leads to acetaldehyde, whereupon this aldehyde can react with more formaldehyde in either single or two-fold aldolisation, thus giving rise to higher aldehydes. These aldehydic substances are subsequently hydrogenated to a series of primary alcohols.

Lead acetate for glycerin determination by the dichromate method. J. KELLNER (Chem. Umschau, 1927, 34, 330).—After describing the preparation of lead acetate according to the I.S.M. 1911 and the purification of a good crude glycerin by the addition of zinc sulphate to the glycerin solution previously made slightly alkaline by caustic soda solution, the effect is described of the basicity of the lead acetate solution on the accuracy of the analytical results. Lead acetate solutions to which varying amounts of lead monoxide have been added are prepared, and from the analytical data recorded it follows that the glycerin found decreases with the increasing basicity of the lead acetate solution. It is not recommended for analytical purposes to use more than 300 g. of lead monoxide with 1 litre of 10% lead acetate solution.

H. M. LANGTON.

Production of vanillin from safrole. A. WAGNER (Chem.-Ztg., 1928, 52, 379).—Vanillin is prepared by heating safrole in an autoclave with absolute alcohol and then with metallic potassium under 16 atm. pressure, the vanillin being isolated from the mixture by a method

similar to that described by Boedecker (B.P. 285,156; B., 1926, 360), whose patent appeared subsequent to the author's work.

E. H. SHARPLES.

Synthesis of phosgene. GIORDANI.—See VII. **Furfuraldehyde as ionising medium.** McKEE and others.—See XI.

PATENTS.

Production of additive products of unsaturated hydrocarbons of the acetylene series. VER. F. CHEM. IND. A.-G., and H. WALTER (B.P. 288,707, 11.1.27).—Acetylene or a homologue (allylene), mixed with excess of vapour of the substance with which it is to react, is passed into a liquid containing a mercury catalyst and mineral acid if desired. Examples are: acetaldehyde from acetylene and steam; ethylidene diacetate or dipropionate from acetylene and acetic acid or propionic acid; vinyl trichloroacetate from acetylene and trichloroacetic acid; α -diisobutoxyethane from acetylene and isobutyl alcohol; α -ethylidenedioxyethane [glycol ethylidene ether] from acetylene and glycol; acetone from allylene and steam or acetic acid; propenyl isobutyl ether from allylene and isobutyl alcohol. C. HOLLINS.

Carrying-out the catalytic oxidation of organic compounds in the gaseous or vapour state. I. G. FARBENIND. A.-G. (B.P. 272,557, 13.6.27. Ger., 14.6.26).—Pure products are obtained in the oxidation of vaporised organic compounds in the presence of non-volatile catalysts, if the reaction mixture coming from the reaction chamber is purified by bringing it into contact with solid adsorbents of large surface (e.g., pumice, earthenware sherds, silica gel, etc.), maintained at a temperature which prevents deposition of the reaction product. The adsorbent must not melt, volatilise, or decompose. It may be impregnated with solutions of salts or bases, or coated with salts or metallic oxides. E.g., commercial naphthalene is oxidised as above, and the gaseous product passed over granulated pumice at 260°. White phthalic anhydride of more than 99% purity is obtained on cooling the purified vapour.

B. FULLMAN.

Manufacture of hydrocarbons of the benzene series. I. G. FARBENIND. A.-G. (B.P. 261,393, 11.11.26. Ger., 12.11.25).—Olefines, particularly ethylene, are converted in two stages into benzene hydrocarbons; in the first stage the olefine is passed at high pressure (e.g., 40 atm.) over a hot catalyst (e.g., charcoal at 400°), and the liquid hydrocarbons so formed are then dehydrogenated above 500°, e.g., by passing the vapour over ammonium molybdate at 600° under 5 atm. pressure or by adding phenols and passing the mixed vapours over tinned iron turnings at 650°. Benzene and its homologues are obtained.

C. HOLLINS.

Manufacture of dinitrohalogen-aryls [halogeno-dinitro-benzenesulphonamides and -benzamides]. I. G. FARBENIND. A.-G. (B.P. 279,134, 18.10.27. Ger., 18.10.26).—Secondary aromatic amines are condensed with 2- or 4-chloro-3 : 5-dinitro-benzenesulphonyl or -benzoyl chloride in presence of water or other diluent and of an acid-binding agent (e.g., excess of the amine) to give the corresponding benzenesulphonamides or

benzamides. 4-Chloro-3 : 5-dinitrobenzenesulphonmethylamide, m.p. 161°, the 2-chloro-isomeride, 4-chloro-3 : 5-dinitrobenzenesulphonbenzylamide, and 4-chloro-3 : 5-dinitrobenzomethylamide are mentioned.

C. HOLLINS.

Manufacture of 1-methyl-2 : 5 : 6-trichloro-3-aminobenzene- [2 : 5 : 6-trichloro-*m*-toluidine]-4-sulphonic acid. I. G. FARBENIND. A.-G. (B.P. 277,372, 12.9.27. Ger., 11.9.26).—2 : 5 : 6-Trichlorotoluene-4-sulphonic acid is nitrated with mixed acid at 70° and the nitro-compound is reduced to 2 : 5 : 6-trichloro-*m*-toluidine-4-sulphonic acid.

C. HOLLINS.

Manufacture of chlorine-substitution products of 1-amino-2 : 4-dimethylbenzene [*m*-4-xylidine] I. G. FARBENIND. A.-G. (B.P. 278,729, 6.10.27. Ger., 6.10.26).—*m*-4-Xylidine is chlorinated in concentrated sulphuric acid in presence of ferric chloride at 0° to give 6-chloro- (m.p. 97—98°) and 2-chloro- (m.p. 47°) derivatives, separable by freezing out; or at the ordinary temperature with more chlorine to give 2 : 6-dichloro-4-*m*-xylidine, m.p. 56—57°, which is isolated as sulphate.

C. HOLLINS.

Manufacture of 2 : 4-dichloro-1-aminoanthraquinone and intermediate products from *m*-dichlorobenzene and phthalic anhydride. I. GUBELMANN, H. J. WEILAND, and O. STALMANN (B.P. 288,884, 15.8.27).—*o*-2' : 4'-Dichlorobenzoylbenzoic acid, m.p. 100—101°, prepared from phthalic anhydride, *m*-dichlorobenzene, and aluminium chloride, is nitrated with mixed acid to give the 5'-nitro-compound, m.p. 174°, which is reduced to the 5'-amino-compound, m.p. 164°. Cyclisation of this with 100% sulphuric acid at 150—160° yields 2 : 4-dichloro-1-aminoanthraquinone, m.p. 205—206°.

C. HOLLINS.

Manufacture of 3 : 4-diaminobenzoyl-*o*-benzoic acids. R. ADAMS, J. M. DAVIDSON, and J. GUBELMANN, ASSRS. to NEWPORT CO. (U.S.P. 1,663,229, 20.3.28. Appl., 13.9.26).—3 : 4-Diaminobenzoyl-*o*-benzoic acid from the corresponding 4-amino-3-nitro-compound by reduction with powdered iron and acetic acid has m.p. 176—177°, and by treatment with concentrated sulphuric acid loses water with the formation of a mixture of 1 : 2- and 2 : 3-diaminoanthraquinone.

A. R. POWELL.

Manufacture of condensation product from olefines and hydrocarbons of the naphthalene series. R. MICHEL, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,667,214, 24.4.28. Appl., 21.6.27. Ger., 4.2.26).—Hydrocarbons of the naphthalene series are treated with olefines at above 50° under raised pressure, in the presence of metal halides having catalytic properties.

L. A. COLES.

Manufacture of diacyl derivatives of naphthalene and acenaphthene. I. G. FARBENIND. A.-G. (B.P. 279,506, 24.10.27. Ger., 23.10.26).—A monoacyl-naphthalene or -acenaphthene additive compound with aluminium chloride, or the ketone itself in presence of aluminium chloride, is condensed with an acyl chloride. Phenyl α -naphthyl ketone with benzoyl chloride gives 60% of 1 : 5-dibenzoylnaphthalene, m.p. 186.5°, and

20% of the 1:8-isomeride, both boiling about 300°/12 mm. 5:6-Dibenzoylacenaphthene, m.p. 143°, and 5:6-diacetylacenaphthene, m.p. 146°, are similarly obtained in yields of 50% and 70–80%, respectively.

C. HOLLINS.

Manufacture of halogen-substituted tertiary aromatic amines. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 288,665, 10.12.26).—Halogenation of tertiary amines of the benzene and naphthalene series in presence of mineral acid (hydrochloric acid) leads mainly to *ortho*-substitution and little *para*. The preparation of 2-chlorodimethyl-*p*-toluidine, b.p. 225–228°, the 3-bromo-compound, b.p. 237–238°, 1-chlorodimethyl- β -naphthylamine, b.p. 185–188°/22 mm., the 1-bromo-compound, b.p. 195°/40 mm. (decomp.; hydrobromide, m.p. 130°), mixed 2- and 4-chlorodimethylaniline, b.p. 206–239°, 2:4-dichlorodimethylaniline, and 3:3-dichloro-4:4-tetramethyldiaminodiphenylmethane, b.p. 276–277°/36 mm., and the 3:3-dibromo-compound is described.

C. HOLLINS.

Manufacture of a 2:3-dichloronitro-1:4-naphthaquinone. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 288,927, 22.11.27).—2:3-Dichloro-1:4-naphthaquinone is nitrated, without appreciable oxidation, by mixed acid (30% HNO_3) at 40–50°, giving dimorphic 2:3-dichloro-5-nitro-1:4-naphthaquinone, m.p. 175°, in 90–95% yield.

C. HOLLINS.

Manufacture of cyclic ketones of the aromatic series [indanones]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 288,441, 26.4.27).— α -Unsaturated carboxylic acids or their hydrogen halide additive products are condensed with aromatic hydrocarbons or their halogen derivatives in presence of aluminium chloride to give α -indanones, or in presence of sulphuric acid to give β -arylpropionic acids which are converted into α -indanones by way of the acid chlorides. Benzene and β -chloropropionic acid with aluminium chloride yield α -indanone, m.p. 40°, toluene gives a mixture of 4- and 6-methylindan-1-ones, chlorobenzene a separable mixture of 4-chloro- (m.p. 91–92°) and 6-chloro- (m.p. 79°) -indan-1-ones. From β -chlorobutyric acid are obtained 3-methylindan-1-one, b.p. 255°, its 4-chloro- (m.p. 47°) and 6-chloro- (b.p. 275°) derivatives, and 3:4:6-trimethylindan-1-one, b.p. 250°. Crotonic acid gives with chlorobenzene the same 4- and 6-chloro-3-methylindan-1-ones, with *m*-dichlorobenzene the 4:6-dichloro-compound, m.p. 77–80°, with *o*-dichlorobenzene an oil, b.p. 200°, and with bromobenzene a solid bromomethylindanone, m.p. 83°, and an isomeride, b.p. 260°. Naphthalene condenses with cinnamic acid in nitrobenzene in presence of 85% sulphuric acid at 45° to give β -phenyl- β -1-naphthylpropionic acid, which is converted by phosphorus pentachloride and aluminium chloride into 1-phenyl- α -naphthindan-3-one

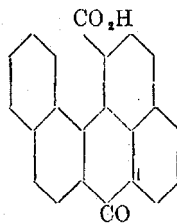
$\text{C}_{10}\text{H}_6 \begin{smallmatrix} \text{CHPh} \\ \diagup \text{CO} \diagdown \end{smallmatrix} \text{CH}_2$, m.p. 140–142°. C. HOLLINS.

Manufacture of alkylpyrazolanthrone. I. G. FARBENIND. A.-G. (B.P. 264,503, 12.1.27. Ger., 13.1.26).—Pyrazolanthrone is alkylated by heating (e.g., at 170°) with alkyl sulphates, alkyl arylsulphonates, or

mixtures of alcohols with sulphuric acid, in presence of acid condensing agents (e.g., sulphuric acid). Methylpyrazolanthrone, m.p. 221–224°, and 6-chloro-2-methylpyrazolanthrone, m.p. 225–227° (from the pyrazolanthrone derived from 1:5-dichloroanthraquinone and hydrazine), give red vat dyes by alkaline fusion.

C. HOLLINS.

Manufacture of new vat dyes and new intermediate compounds. W. CARPMAEL. I. G. FARBENIND. A.-G. (B.P. 288,666, 10.12.26).—1:1'-Dinaphthyl-8:8'-dicarboxylic acid is first treated with an acid condensing agent (e.g., concentrated sulphuric acid at 60°, or zinc chloride in boiling acetic acid) and the resulting benzobenzanthrone-13-carboxylic acid (annexed formula), m.p. 278°, is heated with



potassium hydroxide in alcohol, pyridine, etc. to give a dicarboxylated dibenzanthrone or isodibenzanthrone, which, by cyclisation with an acid condensing agent (concentrated sulphuric acid), yields a greyish-green-blue vat dye. Analogous compounds are obtained from bromo-, dibromo-, and chloro-1:1'-dinaphthyl-8:8'-dicarboxylic acid, the corresponding benzobenzanthronecarboxylic acids melting at 288–290°, above 300°, and 268–269°, respectively.

C. HOLLINS.

Manufacture of bromo-derivatives of 1:1'-dinaphthyl-8:8'-dicarboxylic acid. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 288,957, 10.12.26. Divided from B.P. 288,666; preceding).—Bromination of 1:1'-dinaphthyl-8:8'-dicarboxylic acid in acetic acid gives a yellow monobromo- and, with excess of bromine, an orange dibromo-compound, both melting above 300°.

C. HOLLINS.

Manufacture of an intermediate for [thioindigoid] dyes. I. G. FARBENIND. A.-G. (B.P. 279,436, 14.10.27. Ger., 23.10.26).—3:5-Dichloro-*o*-toluidine is converted by Sandmeyer's reaction into 3:5-dichloro-*o*-tolunitrile, which is hydrolysed with 65% sulphuric acid to give 3:5-dichloro-*o*-toluic acid. The 3-chlorine atom may be replaced by a thiol group and the product used for synthesis of 6:6'-dichloro-4:4'-dimethylthioindigo.

C. HOLLINS.

Manufacture of additive compounds of unsaturated hydrocarbons. H. WALTER, Assr. to VER. F. CHEM. IND. A.-G. (U.S.P. 1,669,384, 8.5.28. Appl., 18.1.27. Ger., 21.1.24).—See B.P. 288,707; preceding.

Manufacture of phenylglycine or compounds thereof. G. IMBERT, Assr. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,668,964, 8.5.28. Appl., 26.10.20. U.K., 2.7.20).—See B.P. 173,540; B., 1922, 170 A.

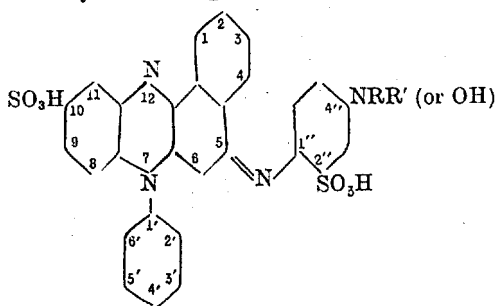
Acetylene as anæsthetic (B.P. 264,826).—See XX.

IV.—DYESTUFFS.

PATENTS.

Manufacture of acid dyes of the phenonaphthasafranine series. J. R. GEIGY A.-G. (B.P. 265,986, 11.2.27. Ger., 13.2.26).—Level blue to green-blue

shades, fast to light and to alkali, are obtained by using azine dyes of the general formula :



containing, if desired, a third sulphonic group and other substituents; the effect of the 2'-sulphonic group is to make the shade greener and to improve the fulling fastness. Diethylisrosinduline-10-sulphonic acid, obtained by oxidation of phenyl- β -naphthylamine together with 4-aminodiethylaniline-2-sulphonic acid, is oxidised in presence of sodium bisulphite to form the 5:10-disulphonic acid, which is condensed with 5-aminoethyl-*o*-toluidine-4-sulphonic acid (from methyl-*o*-toluidine-4-sulphonic acid by coupling with a diazo component and reducing), giving a pure blue wool dye. Diethylisrosinduline-11-sulphonic acid is similarly converted into the 5:11-disulphonic acid and condensed with 4-aminodiethylaniline-3-sulphonic acid for a blue, or with 5-amino-*o*-cresol-4-sulphonic acid for a reddish-blue; 4'-acetamidodiethylisrosinduline-2:5:11-trisulphonic acid (from *p*-acetamidophenyl- β -naphthylamine-7-sulphonic acid and 4-aminodiethylaniline-3-sulphonic acid followed by bisulphite) with 5-aminoethyl-*o*-toluidine-4-sulphonic acid for a greenish-blue. If an acetyl-*p*-diamine is used for the final condensation the product must be hydrolysed in order to attain the desired properties. C. HOLLINS.

Manufacture of acid wool dyes. I. G. FARBENIND. A.-G. (B.P. 282,409, 14.12.27. Ger., 14.12.26).—4-Bromo-1-aminoanthraquinone-2-sulphonic acid is condensed with *p*-aminoacetalkylanilides (e.g., *p*-aminoacetmethylanilide) to give acid wool dyes of redder shade than the non-alkylated compound.

C. HOLLINS.

Manufacture of mordant dyes. J. R. GEIGY A.-G. (B.P. 265,203, 26.1.27. Ger., 28.1.26).—Bright chrome printing reds and violets are obtained by diazotising an aminodiarylmethane, prepared by condensing an arylamine (having a free *o*- or *p*- position) with a chloromethyl-salicylic or -cresotic acid, and coupling with any coupling component. Thus, 4'-amino-4-hydroxy-3-methyldiphenylmethane-5-carboxylic acid, prepared by boiling 5-chloromethyl-*o*-cresotic acid with aniline and water, is diazotised and coupled with α -naphthol-3:6-(or 3:8)-disulphonic acid to give a chrome printing red fast to soap, carbonate, and chlorine, or with 1-(2'-chloro-5'-sulphophenyl)-3-methylpyrazolone for a yellow wool dye (after-chromed, greenish-yellow) suitable also for lakes. The diarylmethanes from 5-chloromethyl-*o*-cresotic acid and *o*-toluidine, cresidine, and metanilic acid are mentioned in other examples; the cresidine compound gives with acetyl-H-acid a chrome printing heliotrope; the

metanilic acid compound, with Schäffer acid a chrome printing orange-yellow. C. HOLLINS.

Dyes and dyeing [soluble leuco-esters of vat dyes]. J. E. G. HARRIS, B. WYLAM, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 288,673, 31.12.26).—The soluble esters of leuco-vat dyes described in prior patents (B.P. 186,057, 202,630, 202,632, 203,681, 212,546, 218,649, 220,964, 231,889, 245,587, 247,787, 248,802, 251,491, 258,626, 260,638, 260,647, and 261,139) are obtained by using pyrosulphuryl chloride, $S_2O_5Cl_2$ (Grignard and Muret, A., 1926, 1113, 1218) in place of chlorosulphonic acid etc. A small amount of water is not harmful in this case as it converts the reagent into chlorosulphonic acid. C. HOLLINS.

Dyes and dyeing [dry dye and leuco-vat dye preparations]. B. WYLAM, J. E. G. HARRIS, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 288,358, 29.12.26 and 13.7.27).—Dye pastes are incorporated with anhydrous or partially hydrated compounds capable of absorbing the water from the dye paste, giving a dry mixture of dye and hydrated substance. The process is applicable to leuco-esters of the Soledon type (in which alkalinity is an advantage) and to dyes in general. Examples are: the leucoflavanthrone ester paste of B.P. 247,787 (B., 1926, 403) with anhydrous sodium carbonate, acetate, sulphate, or disodium hydrogen phosphate; dichloroisodibenzanthrone paste with anhydrous sodium carbonate; 1-amino-2-methylantranthraquinone paste with anhydrous sodium sulphate.

C. HOLLINS.

New azo dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 288,788, 2.3.27).—2-Amino-5-acyl-(oralkyl)amino-4'-hydroxy-3'-carboxydiphenylsulphones (B.P. 267,366; B., 1927, 360) are used as diazo components in azo dyes. The acetyl compound, e.g., is diazotised and coupled with 2-methylindolesulphonic acid (reddish-yellow), acetoacetic 4-sulphoanilide (greenish-yellow), bisacetoacetylthidine (pure yellow), 1-(2':5'-dichloro-4-sulphophenyl)-3-methylpyrazolone (yellow), H-acid (reddish-violet, chromed), methyl- β -naphthylamine-7-sulphonic acid (bluish-red), β -naphthylamine-6- or -7-sulphonic acid (yellowish-red), benzylethylaniline or sulphobenzylethylaniline (yellowish-red), 2-methylamino-6-naphthoic acid (red), γ -acid (coupled acid; bluish-red), methyl- γ -acid (violet), *p*-tolyl- γ -acid (brownish-violet), *N*- β -acetamidoethyl-H-acid (brown). The acetyl group in the diazo component may be replaced by other acyl groups, such as chloroacetyl, oxalyl, carbethoxyl, β -chloroethanesulphonyl, carbamyl, carbonyl, benzoyl, anisoyl, phthaloyl, *p*-toluenesulphonyl, salicyl-5-sulphonyl, 6- or 4-chloro-3-nitrobenzenesulphonyl, etc. The *p*-toluenesulphonyl compound, diazotised and coupled with β -naphthylamine-7-sulphonic acid, gives a pure red chrome dye. 2-Amino-5-acetamido-4'-hydroxy-4-methoxy-3'-carboxy-5'-methyldiphenylsulphone is diazotised and coupled with methyl- γ -acid (violet) or γ -acid (bordeaux-red). 2-Amino-5-acetamido-4'-hydroxy-3'-carboxy-4-methyldiphenylsulphone with γ -acid gives a chrome bluish-red. 2-Amino-5-dimethyl(or diethyl)amino-4'-hydroxy-3'-carboxydiphenylsulphone gives with γ -acid a violet, with methyl- γ -acid a bluish-red, with H-acid a blue.

The 5-formamido-compound diazotised and coupled with γ -acid and hydrolysed with 10% sulphuric acid gives a violet. The 5-acetamido-compound is diazotised and coupled with Cleve's acid, rediazotised and coupled with naphthionic acid, hydrolysed with alkali, diazotised again and coupled with Schäffer acid for a direct blue, becoming reddish-blue on coppering. The various dyes obtained show good fastness properties, are usually little changed in shade on chroming, and are suitable for wool, chrome printing for pigments, and for conversion into chromium-containing dyes by boiling with chromium fluoride. Couplings may be made on the fibre in suitable cases.

C. HOLLINS.

Manufacture of [trisazo direct] dyes. SOC. CHEM. IND. IN BASLE (GES. CHEM. IND. IN BASEL) (B.P. 267,162, 8.3.27. Switz., 8.3.26).—Trisazo dyes of the type: dehydrothio-*p*-toluidinesulphonic acid \rightarrow resorcinol \leftarrow usual non-hydroxylated middle component (B) \leftarrow arylamine (A), give level violet to brown shades on cotton, some of which are fast to light. Suitable dyes may be further diazotised on the fibre and developed. Examples are: (i) B = Cleve's acid, A = sulphanilic acid (brown), aniline (red-brown), *p*-aminoacetanilide (hydrolysed, red-brown; developed with β -naphthol, violet-brown), Cleve's acid (brown-violet), or *p*-nitro-aniline-*o*-sulphonic acid (yellow-brown); (ii) B = α -naphthylamine, A = β -naphthylamine-4 : 8-disulphonic acid (violet-brown).

C. HOLLINS.

Production of [chrome]-azo dyes containing two hydroxynaphthalene nuclei. F. STRAUB and H. SCHNEIDER, ASSRS. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,669,069, 8.5.28. Appl., 7.4.26. Switz., 25.4.25).—See B.P. 251,266; B., 1926, 702.

Manufacture of trisazo dyes derived from dehydrothio-*p*-toluidinesulphonic acid and resorcinol. B. MAYER and J. GRIMMER, ASSRS. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,667,327, 24.4.28. Appl., 24.2.27. Switz., 8.3.26).—See B.P. 267,162, preceding.

Vat dyes (B.P. 288,666).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Attack of cotton by mineral oils at higher temperatures. H. STÄGER (Helv. Chim. Acta, 1928, 11, 377—386).—A comparison is made of the attack of cotton wool by turpentine and transformer oil mixed with various organic acids. The results indicate that the disintegration of the insulation of transformers is not due to acids formed by oxidation of transformer oil, and support the view that interfacial forces are the important factor.

F. J. WILKINS.

Tensile testing of single wool fibres. P. KRAIS (J. Text Inst., 1928, 19, 32—36 T).—The "Deforden" apparatus for measuring the tensile strength, breaking extension, and torsional resistance of single fibres is described in detail, and examples of its application to technical problems are given. There is no difference in strength between wool and "bristly" hairs of the same diameter, but the latter have the higher percentage extension at the breaking point.

B. P. RIDGE.

"Knight test" for feathers. H. F. KNIGHT (Analyst, 1928, 53, 278—279).—To determine the degree

of cleanliness of feathers, a sample (20 g.) is shaken with 1 litre of distilled water, left with occasional shaking for 1 hr., pressed to the side of the bottle, and to 100 c.c. of extract filtered through glass wool is added 1 c.c. of dilute sulphuric acid (1 : 5), and 0.1*N*-potassium permanganate is run in from a burette two drops at a time until the pink colour persists for 1 min. The standard adopted by the Air Ministry is 40 pts. of oxygen absorbed per 100,000, but 15 pts. per 100,000 both for oxygen and chlorine are easily obtained by proper purification.

D. G. HEWER.

Influence of preliminary treatment with alkali in the preparation of cellulose by means of gaseous chlorine. F. GIORDANI (Annali Chim. Appl., 1928, 18, 87—90).—Mutti and Venturi (B., 1927, 904) found that, when esparto is treated successively with sodium hydroxide solution and gaseous chlorine, the yield of cellulose decreases continuously as the concentration of the alkali is increased, whereas the α -cellulose content at first increases and then decreases, the maximum occurring with 4% sodium hydroxide solution. The author's results (B., 1927, 627) show, on the other hand, that the cellulose yield at first increases and then diminishes as the strength of the alkali is increased, the maximum being obtained with 3% sodium hydroxide. Subsequent experiments indicate that the results obtained depend on the relative proportions of alkali and chlorine employed, and further work in this direction is in progress.

T. H. POPE.

Utilisation of "Kaoliang" stalk. I. Digestion of the stalk by the soda process and effect of preliminary treatment on the digestion. R. YAMAMOTO (J. Cellulose Inst., Tokyo, 1928, 4, 53—66).—A relatively easy-bleaching soda pulp can be obtained from the stalk of "Kaoliang" (*Andropogon Sorghum*, Broth.) if the material is pretreated with hot water. Thus, by digestion first with water at 100—120° for 0.5—1 hr. and then, after removal of the aqueous liquor, with 12% of caustic soda at 130—140° for 3—4 hrs., a 46% yield of light brown pulp was obtained which bleached with 2.97% of available chlorine giving a 36% yield of bleached pulp. It is estimated that the annual production of stalk is about 14,400,000 tons.

D. J. NORMAN.

Cellulose fibre. FEUCHTER.—See XIV.

PATENTS.

Treatment of silk. J. C. MACKEY and J. S. KAUFMAN, ASSRS. to VAN RENSSLAER LANSINGH (U.S.P. 1,666,501, 17.4.28. Appl., 19.8.25).—Silk is weighted with tin phosphotungstate. The weighted silk shows the same properties as regards dyeing and printing as does unweighted silk.

D. J. NORMAN.

Degreasing of raw wool and other textile materials. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 287,230, 16.12.26).—*cyclo*Hexanone or a homologue thereof is used either alone or in admixture with other water-soluble organic solvents. Any solvent remaining in the extracted wool may be washed out with water and recovered.

D. J. NORMAN.

Treatment of animal fibres. I. G. FARBENIND. A.-G. (B.P. 264,146, 22.12.26. Ger., 7.1.26).—Sulphite-cellulose waste liquor, or a salt of ligninsulphonic acid,

is used for the protection of animal fibres during treatment with solutions containing chromium compounds for purposes other than dyeing or mordanting (cf. B.P. 188,632; B., 1923, 652 A). D. J. NORMAN.

Disintegration of vegetable fibre bundles. I. G. FARBENIND. A.-G. (B.P. 271,524, 23.5.27. Ger., 21.5.26).—The material is treated, before or after retting, for about $\frac{1}{4}$ hr. at 10–15° with a solution containing a dialkyl-naphthalenesulphonic acid or a salt thereof (0.1–0.5%), optionally with addition of emulsifying agents and other desizing agents. D. J. NORMAN.

Electrically insulating composition of matter for the construction of partitions etc. I. G. FARBENIND. A.-G. (B.P. 280,178, 11.10.27. Ger., 6.11.26).—Asbestos or other fibrous inorganic material, optionally in admixture with inorganic fillers, is impregnated before or after moulding with a solution containing nitrocellulose, a softening agent (preferably a phenol ester of phosphoric acid), and natural and/or artificial resins. The product is non-hygroscopic and does not become conductive in the event of the combustible material therein coking. D. J. NORMAN.

Manufacture of cellulose esters. I. G. FARBENIND. A.-G. (B.P. 268,289, 14.12.26. Ger., 24.3.26).—Improved results are obtained in the manufacture of cellulose esters if the cellulose is pretreated with lactic acid, preferably of about 80% strength. *E.g.*, 1 pt. of cotton is stirred with 20 pts. of 80% lactic acid for about 3 hrs. at 80°. The treated material is then centrifuged, washed, and dried. D. J. NORMAN.

Production of viscose silk fabrics. S. TODA (B.P. 287,807, 22.4.27).—Freshly coagulated viscose threads are loosely twisted (not more than 50 twists per in.) and are then starched and woven into fabric, in which form they are desulphurised and bleached. This procedure permits the filaments to swell during desulphurisation, whereby the covering power of the yarn is increased. D. J. NORMAN.

Production of acetylcellulose compounds and the artificial silk, bands, films, and other artificial textile products, plastic masses, etc. manufactured therefrom. BRIT. ENKA ARTIFICIAL SILK CO., LTD., Assecs. of N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (B.P. 263,771, 29.11.26. Holl., 24.12.25).—Cellulose is acetylated in a bath the water content of which has previously been reduced by allowing the acetic acid-acetic anhydride mixture to remain, with the exclusion of water or water vapour, for at least 2 hrs. before and 1 hr. after the addition of the catalyst. Since the viscosity of the cellulose acetate increases as the quantity of water present in the reaction mixture is reduced, it is possible, by using an acetylating bath which is practically anhydrous, *i.e.*, which has been kept for about 10 hrs., and adjusting the moisture content of the cellulose, to control to a large extent the viscosity and other properties of the final product. D. J. NORMAN.

Manufacture of sheets or films of compositions containing cellulose esters or ethers. SPICERS, LTD., and H. J. HANDS (B.P. 287,635, 23.12.26).—The last traces of volatile solvent are removed from freshly-prepared cellulose ester or ether films by passing the

film under slight tension through a tunnel or similar device into which low-pressure steam is continuously introduced in sufficient quantity to maintain a warm, damp atmosphere without substantially wetting the film. This procedure prevents subsequent cockling and distortion, and removes any electrical charge which may have accumulated on the film during manufacture. D. J. NORMAN.

Cellulosic materials and products obtained therefrom. BRIT. CELANESE, LTD., H. DREYFUS, and C. I. HANEY (B.P. 288,657, 8.10.26).—Cellulosic materials are made more reactive to esterifying agents by pre-treating them with small quantities, *e.g.*, 10–30% on the weight of cellulose, of formic acid of 50–100% (preferably 70–90%) strength, optionally in admixture with acetic acid. The solution may conveniently be applied by spraying, and the impregnated material is kept for a period up to 12 hrs. at the ordinary temperature or for a shorter time at a raised temperature. It is unnecessary to remove the formic acid before esterification, but an extra quantity of acetic anhydride must be added to the acetylating bath to compensate for the wastage due to the presence in the cellulose of formic acid and any water associated therewith. D. J. NORMAN.

Treatment of yarns, fabrics, films, etc. H. DREYFUS (B.P. 284,798, 12.11.26. Addn. to B.P. 281,084; B., 1928, 227).—The following compounds may be used, either alone or in admixture, as swelling agents in the weighting of organic substitution derivatives of cellulose by the process of the earlier patent: thiocarbimides, cyanates, or carbimides, particularly those of the alkali metals and ammonium; urea, urethanes, thiourea, thiourethanes, guanidine, or their alkyl, aryl, or aralkyl substitution products. D. J. NORMAN.

Utilisation of lignin. R. GRIESSBACH and J. EISELE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,666,696, 17.4.28. Appl., 18.10.27. Ger., 22.10.26).—Material rich in lignin is extracted with an organic solvent containing a halogen. A. R. POWELL.

Manufacture of paper which possesses properties closely resembling those of hand-made paper. C. MOES (B.P. 282,620, 30.5.27. Holl., 24.12.26).—A continuous web of paper is made on the substantially uninterrupted surface provided by a number of separate, wire-cloth covered suction boxes mounted on a moving endless belt. The suction boxes and couch roll are given a lateral shake as a whole, relative displacements of the boxes during the formation of the sheet being prevented. The finished web shows weak cross-zones, and when torn at these points a "deckle-edge" effect is obtained. D. J. NORMAN.

Proofed paper. J. REID, Assr. to SCUTAN CO. (U.S.P. 1,667,691, 24.4.28. Appl., 26.11.26).—Strong, close-grained, flexible paper is passed through a bath of a bituminous proofing agent of relatively high m.p. (above 121°). Impregnation is limited to the surface layers of the paper by adjusting the duration of the treatment and maintaining the bath at such a temperature that the proofing material is liquid but still viscous. D. J. NORMAN.

Manufacture of artificial textile products. H. DREYFUS (U.S.P. 1,669,158, 8.5.28. Appl., 26.5.23. U.K., 6.7.22).—See B.P. 209,125; B., 1924, 213.

Apparatus for the manufacture of [twisted] yarns or threads. BRIT. CELANESE, LTD., W. A. DICKIE, and R. MARTIN (B.P. 289,321, 22.10. and 8.12.26).

Spinning-pump arrangements for machines for manufacturing artificial textile thread. H. KINDERMANN (B.P. 289,497, 23.11.26).

Apparatus for treatment [jigging] of fabrics in industrial processes. WHITEHEAD & POOLE, LTD., and J. WAGSTAFF (B.P. 289,616, 5.4.27).

Increasing the capacity of drying installations [for paper-making]. AKTIEBOLAGET SVENSKA FLÄKTFABR., ASSECS. of J. G. OLSSON (B.P. 281,339, 28.11.27. Swed., 29.11.26).

Briquetting of fuels etc. (B.P. 274,876). **Coated paper** (B.P. 273,281). **Bituminous emulsions** (B.P. 288,821).—See II. **Fatty material from emulsions.** (B.P. 289,350).—See XII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Affinity of amidated and pyridinated fibres for acid substances. P. KARRER and S. C. KWONG (Helv. Chim. Acta, 1928, 11, 525—529).—The amounts of seven organic and inorganic acids and five acid dyes which combine with amidated and pyridinated cotton fibres (cf. B., 1926, 659) have been determined. Using an amidated fibre of nitrogen content 0.71%, about 0.05 g.-equiv. of each acid is fixed, whilst the value for a fibre of nitrogen content 0.21% is 0.015—0.02, even if the acid bath is varied from 0.1N to 0.5N. Both amidated and pyridinated fibres combine with acid dyes. There appears to be no relationship of the amount fixed and the nitrogen content of the fibre as is the case with acids. H. BURTON.

PATENTS.

Stabilising of bleaching liquors [soap powders etc.]. T. BENCKISER, A. REIMANN, and A. REIMANN, JUN. [J. A. BENCKISER], and F. DRAISBACH (B.P. 289,156, 21.1.27).—The action of alkali pyrophosphates in stabilising solutions containing peroxides and per-salts is improved by adjusting the hydrogen-ion concentration of the solution to p_{H} 7—8.5 in the absence of soap, or to p_{H} 7—10 in the presence of soap, by adding mineral acids or acid salts to the solutions, or acid salts to the dry mixtures used in preparing the solutions. L. A. COLES.

Production of resists on animal fibres. K. DAIMLER, C. E. MÜLLER, and H. GÄRNER, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,668,144, 1.5.28. Appl., 12.2.26. Ger., 20.2.25).—See B.P. 248,007; B., 1927, 628.

Spool or cop cores for spool or cop dyeing. J. ANNICQ (B.P. 289,708, 10.10.27).

Dyeing (B.P. 288,358 and 288,673).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Modified circulation system improves operation of chamber [sulphuric acid] plant. E. L. LARISON

(Chem. Met. Eng., 1928, 35, 229).—In operating three Gay Lussac towers in series with regard to acid feed as well as gas, a larger amount of acid is circulated through these towers than is fed to the Glover tower, the excess simply overflowing from one tank to another at the bottom. The best conditions for Glover operation (restricted feed of nitrous vitriol) are thus combined with a desirably ample feed for complete absorption of nitrous gases in the Gay Lussac towers. A considerable economy in nitre was obtained.

C. IRWIN.

Development of the process and equipment for the manufacture of chemically pure acid by electrical distillation. **Hydrochloric and nitric acids.** C. M. HOFF (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 24 pp.).—The development of an improved commercial process for the production of chemically pure hydrochloric acid by distillation of the crude commercial acid is outlined. In the process finally adopted and operated by the Grasselli Chemical Co. a continuous distillation system using electrical heating is employed and has led to considerable reduction of operating costs and breakage, whilst the working conditions and the quality of the product have improved. The crude acid is fed at a controlled rate into a granite retort, and in it are submerged two graphite electrodes between which an A.C. is passed. These electrodes are plates drilled with several small holes to ensure easy circulation of the acid, and if the current density does not exceed about 300 amp./ft.², there is no evidence of electrolysis or disintegration of the electrodes. Granite was adopted as the material for retort construction after unsuccessful trials with earthenware, glass, and silica. To the top of the retort are connected four pyrex glass condensers by way of an earthenware manifold with four outlets luted with phosphoric acid. From the manifold the condensers slope upwards for a short distance in order to permit of some refluxing and then bend over forming a long vertical section down which the acid passes into a collecting vessel and thence through a "gravity jar" into carboys. At given power input the concentration of the product, as shown by the gravity jar, can be readily controlled by adjusting the rate of flow of crude acid into the retort. A loose packing of glass wool in the end of the condenser joining the retort was found effective in preventing contamination of the distillate with traces of iron. About one seventh of the acid entering the retort is run off to waste in order to keep down the concentration of impurities. One kw.-hr. produced as much chemically pure acid as 3.45 kg. of coal by the old process. Details of plant construction are described and the application of the process, with certain modifications as to materials of construction, to the distillation of crude nitric acid is discussed. H. J. T. ELLINGHAM.

Modification of Ridsdale's method for determining phosphoric acid. A. S. DODD (Analyst, 1928, 53, 276—278).—Certain modifications of the details given by Cameron and Dow (cf. B., 1927, 919) are suggested, chief among which is the use of a simple wash-bottle device, fitted with cotton wool pad, for siphoning off supernatant liquid during the washing of the precipitate. The whole determination is made in one

vessel, and centrifuging is unnecessary; strong nitric acid with 2 vols. of water is used in place of acid of d 1.20; 0.5*N*-sulphuric acid replaces the nitric acid and *N*-sodium hydroxide the 0.5*N*-alkali; and 5 c.c. of dilute nitric acid (1 : 3) is used instead of 4 c.c. of nitric acid (d 1.20).
D. G. HEWER.

Prediction of boiling points of concentrated caustic-salt [sodium hydroxide-sodium chloride] solutions. F. W. ADAMS and C. W. RICHARDS (Ind. Eng. Chem., 1928, 20, 470–471).—The b.p. of sodium hydroxide solutions of varying concentrations and saturated with sodium chloride were determined for absolute pressure of 50–760 mm. of mercury. If the temperature of the solution, whether of sodium hydroxide or of the latter with sodium chloride, is plotted against the temperature of pure water for varying pressures, a series of parallel straight lines is obtained corresponding to different concentrations. This indicates that within this range Dühring's rule holds good, and if the b.p. at two pressures is known it can be predicted for any other pressure. The data are required for the design of multiple-effect evaporators.

C. IRWIN.

Reduction of sodium sulphate with carbon. P. P. BUDNIKOV and A. N. SYSOIEV (Z. anorg. Chem., 1928, 170, 225–232).—In the reduction of sodium sulphate with carbon at 800–950°, the main reaction is represented by the equation $\text{Na}_2\text{SO}_4 + 2\text{C} = \text{Na}_2\text{S} + 2\text{CO}_2$; carbon monoxide in the gaseous products of reaction appears to be formed by the reaction $\text{CO}_2 + \text{C} = 2\text{CO}$. The formation of carbonyl sulphide, presumably by the reaction $\text{Na}_2\text{S} + 2\text{CO}_2 = \text{Na}_2\text{CO}_3 + \text{COS}$, can also be detected. Further, if hydrocarbons are associated with the carbon, water is produced, which reacts with the sulphide liberating hydrogen sulphide, and forming sodium hydroxide, the latter then combining with carbon dioxide. When the reduction is carried out under a pressure of 15 mm., separation of free sulphur occurs. In an atmosphere of nitrogen at the ordinary pressure, reaction starts at 750–880°, depending on the kind of carbon used, and takes place at first entirely between solids. With sufficient excess of carbon, a yield of up to 95% of sulphide may be obtained in 2–3 min. at 850–1000°. It therefore appears probable that the present hearth process of manufacture might profitably be replaced by a process in which the reaction is distributed over a large volume, *e.g.*, by blowing the finely-divided sulphate into flames of pulverised fuel or carbon monoxide.
R. CUTHILL.

Influence of the "conversion" yield and the temperature of condensation on the purity of the product and on the "condensation yield" in the synthesis of phosgene. F. GIORDANI (Annali Chim. Appl., 1928, 18, 90–96).—The relationships between (1) the proportion, x , of the equimolecular mixture of carbon monoxide and chlorine in the feed gases, (2) the conversion yield, y , which is the fraction of the equimolecular mixture reacting, (3) the condensation yield, ρ , and (4) the gross yield η , when phosgene is produced under different conditions, are considered. As x diminishes, ρ and η also diminish, and the content, τ , of phosgene in the resulting liquid increases slightly;

diminution of y influences appreciably both τ and also the yields, and in practice y should not be below 0.96. A slight excess of carbon monoxide in the initial gases results in greater purity of the phosgene produced. As the temperature of reaction falls, the purity also falls, but ρ and η increase rapidly and tend to assume an asymptotic course. In practice, no lower temperature than -20° should be employed, except in cases where the presence of chlorine in the product is unimportant.
T. H. POPE.

Colloidal behaviour of lime. RAY and MATHERS.—See IX. **Equilibria in reduction of ferric oxide.** FURNAS and BROWN.—See X. **Iron in red lead.** HEINRICHS.—See XIII.

PATENTS.

Manufacture of hydrocyanic acid from crude cyanides. G. H. BUCHANAN, Assr. to AMER. CYANAMID Co. (U.S.P. 1,667,838, 1.5.28. Appl., 25.3.24).—Crude cyanides containing alkaline-earth compounds are treated with water, any hydrocyanic acid formed being removed from the zone of reaction before any objectionable quantity of azulmic compounds are formed.

H. ROYAL-DAWSON.

Manufacture of phosphoric acid. I. HECHENBLEIKNER, Assr. to CHEM. CONSTRUCTION Co. (U.S.P. 1,667,549, 24.4.28. Appl., 1.4.24).—Dilute phosphoric acid is added to phosphate rock during grinding, and the overflow, which carries in suspension the finer particles of rock, is treated with sulphuric acid.

L. A. COLES.

Filter plate. Cellular silica product. II. L. WATSON, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,669,362—3, 8.5.28. Appl., [A] 31.1.24, [B] 15.8.27).—(A) Particles of silica are caused to adhere to one another without the aid of a binder and to expand into an intumescent, cellular condition. (B) Highly porous silica is prepared by heating crystalline silica at about 1700°, cooling it to below 230°, impregnating the product with a decomposable binder, and reheating rapidly to about 1750°.

F. G. CROSSE.

Ammonia synthesis process and catalyst. A. T. LARSON, Assr. to LAZOTE, INC. (U.S.P. 1,667,322—3, 24.4.28. Appl., [A] 30.7.25, [B] 16.2.26).—Particles comprising an intimate mixture of (A) iron oxide, potassium oxide, and magnesium oxide, or (B) iron oxide, an alkali oxide, and an alkaline-earth oxide, are treated at 300–700° with a gas containing hydrogen and free from catalyst poisons.

L. A. COLES.

Purification of sodium sulphide. R. BOTSON and SOC. IND. DES APPL. CHIM., SOC. ANON. (I.N.D.A.C.) (B.P. 289,354, 14.11.27).—Commercial sodium sulphide is liquefied in a digester by pressure steam, and purified by the addition of 1% of terpinolene. After dilution with water it is decanted and filtered. The density of this liquor is then raised to 1.15 by means of caustic soda (d 1.32) and the product filtered, concentrated, and crystallised out to give pure sodium sulphide suitable for photographic work.
W. G. CAREY.

Production of nitrate of lime. APPAREILS & EVAPORATEURS KESTNER (B.P. 289,370, 13.3.28. Fr., 6.5.27. Addn. to B.P. 279,037; B., 1928, 230).—Sandstone spheres, hollowed or pierced, are added with

the limestone in the proportion of 2% by vol. in order to keep constant the resistance offered by the charge to the nitric acid and gaseous currents, and an inclined grating is provided at the base of the tower to facilitate their removal.

W. G. CAREY.

Manufacture of calcium arsenate. W. C. PIVER (U.S.P. 1,667,490—1, 24.4.28. Appl., [A] 18.1.24, [B] 5.7.24).—(A) Arsenic acid solution of suitable density is added with stirring to dry calcium hydroxide, prepared by adding to calcium oxide just sufficient water to hydrate it, at such a rate that the heat evolved drives off any water present, yielding a dry powdered product. (B) Arsenic acid is added to partially hydrated calcium oxide containing sufficient free calcium oxide to combine with the free water, yielding a dry commercial product.

L. A. COLES.

Manufacture of salts of the alkaline-earth metals from alkaline-earth sulphides. J. Y. JOHNSON.

From I. G. FARBENIND. A.-G. (B.P. 289,196, 14.2.27).—Alkaline-earth sulphides are trituated, *e.g.*, in an edge-runner mill, with the ammonium salt of the desired acid, the mixture being heated and/or sprayed with water during the process.

L. A. COLES.

Production of potassium ferrocyanide. G. H. BUCHANAN and C. M. HULINGS, Assrs. to AMER. CYANAMID Co. (U.S.P. 1,667,839, 1.5.28. Appl., 27.3.24).—Calcium ferrocyanide is added in excess to a ferrocyanide mother-liquor, then commercial potassium chloride. The insoluble constituents are removed from the solution and treated with sufficient potassium carbonate to decompose any double salt of potassium and calcium ferrocyanide, but insufficient to react with any calcium sulphate present. Potassium ferrocyanide is then obtained from the filtered solution.

H. ROYAL-DAWSON.

Manufacture of copperas [ferrous sulphate crystals]. H. C. STEWART, Assr. to WESTMORELAND CHEM. & COLOR Co. (U.S.P. 1,667,693, 24.4.28. Appl., 3.5.23).—A solution of ferrous sulphate (*d* 1.32—1.38) is allowed to fall in a thin stream against a transverse current of air to atomise the solution and effect crystallisation of the contained salt.

A. R. POWELL.

Manufacture of lead salts [from lead chloride]. S. C. SMITH (B.P. 289,105, 20.10.26).—An aqueous suspension of "active" lead chloride, prepared by pouring a hot solution saturated with sodium chloride and lead chloride into about an equal volume of cold water and collecting and washing the precipitated lead chloride, is treated at the boil with the acids of arsenic, antimony, chromium, tungsten, molybdenum, or uranium for the production of the lead salts of these acids.

L. A. COLES.

Conversion of [recovery of lead compounds from] lead sludge. J. B. HILL, Assr. to ATLANTIC REFINING Co. (U.S.P. 1,667,550, 24.4.28. Appl., 16.7.27).—The sludge is treated with an alkali hydroxide solution in the presence of a gas containing oxygen.

L. A. COLES.

Zinc oxide catalysts. G. BLOOMFIELD, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,668,838, 8.5.28. Appl., 21.6.26. Renewed 12.10.27).—Zinc oxide is treated with a solution of ammonium nitrate, and the product ignited.

F. G. CROSSE.

Production of basic aluminium sulphate. D. B. BRADNER, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,663,435, 20.3.28. Appl., 19.3.25).—Finely-ground bauxite is digested at 90—105° with a solution of aluminium sulphate containing 7—12% Al_2O_3 and with a reducing agent (*e.g.*, aluminium dross or sulphur dioxide) which reduces the iron to the ferrous condition. The resulting solution contains about 11% of Al_2O_3 , of which 8.5% is present as aluminium sulphate and 2.5% as colloidal hydroxide.

A. R. POWELL.

Preparation of oxygen-evolving preparations suitable for use in respiratory apparatus etc. DEUTS. GOLD- u. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 280,554, 9.11.27. Ger., 10.11.26).—Alkali peroxides are stirred and cooled with small quantities of atomised water (preferably 6—7%), oxygen catalysts (*e.g.*, oxides of iron, manganese, etc.) having been incorporated before or after conversion into peroxides.

W. G. CAREY.

Production of gas. H. H. SMITH, Assr. to MINERALS SEPARATION NORTH AMERICAN CORP. (U.S.P. 1,667,272, 24.4.28. Appl., 14.9.22).—A gas consisting mainly of hydrogen sulphide is produced by passing steam and a restricted amount of air through a heated mixture of carbon and a sulphur-bearing material.

F. G. CLARKE.

Production of mixtures of hydrogen and nitrogen. W. GAUS and W. WILD, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,666,694, 17.4.28. Appl., 5.2.25. Ger., 14.11.22).—A gas producer is operated with steam and with two different oxygen-nitrogen mixtures, one richer and one poorer in oxygen than atmospheric air and both derived from the rectification of liquid air. The proportions of steam and the two gas mixtures passed over the incandescent fuel are so regulated that the gas issuing from the producer consists of a mixture of 1 vol. of nitrogen to 3 vols. of hydrogen and carbon monoxide together, and that the producer is kept in continuous operation without intermediate heating periods. The producer gas is then subjected to treatment adapted to substitute hydrogen for carbon monoxide, and the carbon dioxide formed in this operation is removed by known methods.

A. R. POWELL.

Apparatus suitable for the gradual production of gases. W. E. KEMMERICH (U.S.P. 1,668,371, 1.5.28. Appl., 1.4.25).—A receptacle, half full, for example, of hydrochloric acid and infusorial earth, has a close-fitting cover which comprises an annular trough having overflow passages leading into the receptacle and supports a central vertical tube the upper end of which is closed. *E.g.*, the tube with its lower end protruding into the receptacle may contain potassium permanganate and sand; water poured into the trough will cause slow evolution of chlorine.

F. G. CLARKE.

Regeneration or reactivation of catalysts. M. LARSSON, Assr. to PHOSPHORUS HYDROGEN Co. (U.S.P. 1,668,539, 1.5.28. Appl., 11.1.26. Swed., 29.9.25).—Catalysts used in the decomposition of steam by means of phosphorus are regenerated by treatment with hydrogen at such temperatures that the inert coating is converted into a substance which will react with steam.

F. G. CLARKE.

Storage of volatile liquids (B.P. 263,775).—See I.
Hydrogen from hydrocarbons (B.P. 288,662).—See II.

VIII.—GLASS; CERAMICS.

Preparation of water-glass [sodium silicate]. K. VON REIBOLDT (Chem.-Ztg., 1928, 52, 345—346).—Practical hints are given for improving the quality and output of fused sodium silicate and for converting the product into water-glass. A. R. POWELL.

Iron in red lead. HEINRICH. —See XIII.

PATENTS.

Manufacture of composite glass. H. J. MALLABAR, and NON-INFLAMMABLE FILM CO., LTD. (B.P. 288,782, 24.2.27).—A relatively thin layer of cellulose acetate, capable of being cast from solution in continuous rolls, is fastened by gelatin between sheets of glass. Bonding is secured by drying the gelatin layer with which the surfaces are coated, treating with a softening liquid, and uniting under pressure. Before applying the gelatin layer to the surface of the cellulose acetate, the latter is suitably treated, as by partial hydrolysis or with a solvent liquid, whilst the glass surface is prepared by coating with a gelatin solution containing a hardening agent. The gelatin layer for bonding is made by dissolving this substance in a water-acetic acid-methyl alcohol-acetone mixture. The glass may be rendered colourless by imparting a bluish-violet tint to the cellulose acetate, gelatin, or glass. A. COUSEN.

Enamels based upon barium and strontium compounds and adapted for the enamelling of cast and wrought iron, copper, and other metals. SOC. MIGEOT FRÈRES & ARNOULD (B.P. 273,260, 19.5.27. Fr. 26.6.26).—A transparent enamel for applying to metals previously coated with opaque enamels is free from lead and titanous acid, and contains 5—50% of baryta or strontia. A suitable composition contains 21% of borax, 11.25% of boric acid, 25% of baryta, 8.75% of felspar, 11% of alkali, 11.5% of lime, 10% of zinc oxide, and 1.5% of alumina. Fluorine compounds and colouring agents may also be added. L. A. COLES.

Glazes for the manufacture of ceramic ware. J. W. MELLOR, and CERAMIC PATENT HOLDINGS, LTD. (B.P. 288,679, 4.1.27).—A "leadless" glaze, capable of maturing at low temperatures, consists of approximately 2 pts. of Cornish stone (or its equivalent) and 1 pt. of borocalcite or an insoluble or very sparingly soluble calcium borate, and it contains no artificially fritted material. Other constituents, designed to improve dipping properties and tendency toward settling, may be added. A. COUSEN.

Manufacture of ceramic products. L. KERN (U.S.P. 1,666,936, 24.4.28. Appl., 13.10.25).—A mixture of sand, hydrated lime, and silicic acid is moulded under pressure and treated under pressure with a gas which hardens the product by reacting with it.

F. G. CLARKE.

Drying of sanitary pipes and other clay goods. G. O. LAWTON, S. DOWTHWAITE, and ALBION CLAY CO., LTD. (B.P. 289,225, 15.3.27).

IX.—BUILDING MATERIALS.

Colloidal behaviour of lime. K. W. RAY and F. C. MATHERS (Ind. Eng. Chem., 1928, 20, 475—477).—The plasticity of a lime putty made by slaking quicklime with excess water as compared with a putty made by adding water to the dry hydrate is believed to be due to the formation of an irreversible colloid during slaking. This colloid is destroyed during the drying of the hydrate and not again formed. Exceptional cases are due to the presence of magnesia which is not completely slaked during hydration. In order to confirm this hypothesis, the variation in the rates of settling of suspensions of different limes under an electric field was determined. Considerable evidence of the existence of electrically charged particles in plastic limes was obtained. The effect of these colloid particles in conferring plasticity or non-drying qualities on putties may be explained by supposing the charges to hold a film of water round the particles more firmly. It is shown that this must be the case if the electrical charges are due to the selective absorption of ions, an osmotic pressure being set up against which the drying must be performed.

C. IRWIN.

Waste heat in Portland cement manufacture. H. POOLEY, JUN. (Engineering, 1928, 125, 497—498, 562—563).—In view of the greater uniformity of the final product, Portland cement is being made increasingly by means of the "wet process," though the recuperation of heat obtainable on the average wet-process plant is much less than that necessary to run the mechanical side of the works, in contrast to conditions prevailing on the "dry-process" system. Methods which tend to eliminate the wastage of a large amount of heat leaving the kiln at about 450° include those which artificially increase the temperature of the exit gases until they contain the heat necessary for the works' plant, or, in the reverse direction, cooling the gases to a greater extent by useful work before leaving the kiln. Thus, a rotary kiln normally about 200 ft. long might be shortened by 40 ft., and the waste gases, now at about 700°, provide all heat required by the power plant; the coal consumption would be reduced from 42% (expressed as a percentage of the cement produced) to a comparative value of 35%. The effective drying zone of the wet-process kiln can be increased by, e.g., spraying the slurry and generating the necessary power independently, the estimated total coal consumption then being 37%. In a process in which the slurry is dewatered from, say, 38% to 20% of contained water, as obtains at the plant of the Ford Motor Co., the consumption of coal may be expected to be 29—30% of the cement produced. Other operating disadvantages may then be expected, though possibly the atomiser system would show the greater relative advantage when the water content of the slurry is high. Conservation of heat from the cooling clinker is best effected by a current of air which later is used in the kiln combustion, the cooling taking place in what is essentially an extension of the lower end of the kiln—a practice already in common usage. C. A. KING.

PATENTS.

Production of unsintered binding means [hydraulic cement] from oil shale and limestone.

O. TETENS (U.S.P. 1,665,993, 10.4.28. Appl., 29.12.24. Ger., 21.5.22).—Raw shale is mixed with limestone, the mixture distilled to remove volatile oils, the residual mass of coke and lime burnt in the same kiln, and the product ground (cf. U.S.P. 1,536,165; B., 1925, 549).

A. R. POWELL.

Cement compositions. E. C. PALM and H. T. THORSSON (B.P. 288,903, 28.9.27).—The compositions comprise Portland cement and a powdered mineral containing hydrated magnesium silicate, *e.g.*, chlorite or serpentine, but practically free from asbestos.

L. A. COLES.

Manufacture in the cold state of tiles for walls and slabs for floors. A. FEIST and F. L. KOPP (B.P. 288,867, 18.7.27).—A hardening agent, *e.g.*, a mixture of bituminous material with silicofluorides, is incorporated into a pasty mixture of magnesium chloride lye with an intimate mixture of finely-ground burnt magnesite, sand, talc, and colouring material, and the product is allowed to harden in moulds.

L. A. COLES.

Coated paper (B.P. 273,281).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Equilibria in the reduction of ferric oxide. C. C. FURNAS and G. G. BROWN (Ind. Eng. Chem., 1928, 20, 507—510).—The authors have obtained thermal data for iron and its oxides for temperatures up to 800°, including heats of transformation, absolute entropies, and heat capacities. From these, with the help of extrapolation, the relation of heat of formation to temperature for the two oxides is shown graphically up to 1800°, and that for carbon dioxide, carbon monoxide, and water vapour is also expressed graphically. Equilibrium constants were then obtained for the various possible reactions in the blast furnace from the relation $\Delta F^\circ = -RT \log k$ at temperatures from 100° to 1500°. They indicate that the reduction of ferric oxide with solid carbon can scarcely take place below 1000°, and that whilst the oxidising effect of steam on iron decreases with rise of temperature, the oxidising effect on magnetic oxide of iron increases. Very low dissociation pressures of ferric oxide are indicated even at 1500°. The effect of energy changes which may occur in the formation of solid solutions is not known, and no thermal data are available for ferrous oxide.

C. IRWIN.

Application of the nitridation of certain special steels. L. GUILLET (Compt. rend., 1928, 186, 1177—1180; cf. B., 1926, 410).—Chromium-aluminium steels hardened by nitridation have been used successfully for the cylinders of internal-combustion engines and for propeller-shafts. In the former case wearing is reduced to a minimum by the polishing effects of the pistons, and the normal oil consumption of the motor remains at its initial figure even after prolonged usage. In the latter the polished surfaces obtainable have resulted in a gain of 10% for a shaft working at 3000 r.p.m.

J. GRANT.

Deposition of metals on copper from cyanide solutions. I. Separation and determination of small amounts of lead. B. S. EVANS (Analyst, 1928,

53, 267—275).—Lead, especially in the presence of ammonium oxalate, will deposit quantitatively in the cold from cyanide solutions. Tin, antimony, zinc, cadmium, and nickel do not interfere in the cold. The apparatus used is as for the determination of mercury (cf. Evans and Clarke, A., 1926, 704). A 5 g. sample is dissolved in 60 c.c. of nitric acid (*d* 1.2) (or if tin is present 35 c.c. of nitric acid and 25 c.c. of hydrochloric acid are used), excess of acid is boiled off somewhat after dissolution, about 9 g. of potassium bitartrate are added, the solution is cooled, the liquid made slightly alkaline to litmus with sodium hydroxide, and saturated potassium cyanide solution added until the precipitate has re-dissolved, the necessary excess of this reagent being then adjusted (2—3 c.c. for copper, about 5 c.c. for bronze, 10 c.c. for brass and zinc, and 30 c.c. for nickel and cupro-nickel) at the point where the precipitate is almost dissolved. The deposition tube is prepared much as for the determination of mercury. Percolation is carried on for 2 hrs., the lead completely removed from the copper by glacial acetic acid, the liquid treated with dilute sulphuric acid and a little nitric acid, and the lead in the impure lead sulphate determined by matching it as lead chromate solution against a potassium dichromate solution. The effect of small amounts of elements liable to occur in commercial copper was found to be negligible; results for brass are more uneven than for copper or zinc, owing to segregation of the lead; nickel and iron do not interfere, and phosphates exert a slight restraining influence. Lead can by this process be quantitatively separated from at least 10,000 times its weight of copper, 1000 of tin, 5000 of nickel or zinc, 20,000 of sulphuric acid, or corresponding weights of alkali or ammonium sulphates. An adaptation of the process is described for making it universally applicable in the presence of large amounts of tin, phosphates, or mechanically interfering substances. D. G. HEWER.

Melting sterling silver in high-frequency induction furnaces. R. H. LEACH (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 5 pp.).—Data are given for the melting of sterling silver scrap in two types of Ajax-Northrup high-frequency furnace, (1) the type energised by high-frequency current obtained from a Tesla oscillator with a mercury discharge gap, and (2) that energised by high-frequency current produced by a motor generator set of special design. In type (2) the much lower frequency causes a large percentage of the inductive heating to occur in the metal instead of in the crucible, and produces a vigorous stirring action. Large units of this type are now in use. In a unit of 100 kw. rated capacity supplied with single-phase 480-cycle current at 1800 volts and using water-cooling of the coil, a charge of 432 kg. of the silver alloy is melted in 50 min. with an energy input of 115 kw.-hr. Comparative data for operation with an air-cooled coil are given. There are 48 condensers, of which 12 can be thrown in or out of circuit to control the power factor. Operating costs compare favourably with the oil-fired furnaces previously used.

H. J. T. ELLINGHAM.

Detection of zinc in presence of iron. W. H. HAMMOND (Chemist-Analyst, 1928, 17, 14).—For the

detection of zinc in spent electrolyte, 10 c.c. are mixed with 2 c.c. of 85% phosphoric acid and 1 drop of 0.5% copper sulphate solution containing 0.5% of concentrated sulphuric acid. On stirring with 4 drops of a solution containing mercuric chloride (8%) and ammonium thiocyanate (9%), zinc yields a violet precipitate which is compared colorimetrically with that from a solution containing 0.0025% Zn.

CHEMICAL ABSTRACTS.

Behaviour of plating baths and anodes during electrodeposition of chromium. R. SCHNEIDEWIND and S. F. URBAN (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 29 pp.).—The behaviour of chromium plating baths has been studied with special reference to anodic influences. During electrolysis of chromic acid baths containing potassium sulphate and various amounts of chromic hydroxide using steel anodes, the concentration of trivalent chromium increases or decreases so as to approach an equilibrium value, C_e , which, under given conditions of electrolysis, is practically independent of its initial value. During electrolysis iron dissolves from the anodes at a rate which is constant at given current density. Increase of current density or temperature increases C_e , but increase of current density appears to cause a slight decrease in the rate of anode corrosion (g./amp.-hr.). The effect of current density on C_e becomes very small when the former exceeds 5.4 amp./dm.² at the anode. Increasing the sulphate content of the bath increases C_e and the rate of anode corrosion according to practically linear relations. Intermittent electrolysis increases C_e notably without materially affecting the rate of anode corrosion (calculated on actual time of plating). With lead anodes C_e is much lower than with steel under the same conditions, and the value is hardly affected by intermittent electrolysis. At high temperatures lead dissolves more rapidly than iron or steel. The presence of iron or trivalent chromium in the electrolyte does not notably affect the cathodic current efficiency, but it reduces the range of current densities within which satisfactory deposits can be obtained and it decreases the conductivity of the bath. The coating of lead chromate of high resistance which tends to form on lead anodes can be removed by treatment with a saturated sodium chloride solution slightly acidified with hydrochloric acid. The relative merits of lead and steel anodes for chromium plating are discussed.

H. J. T. ELLINGHAM.

Effect of trivalent chromium and iron on chromic acid chromium plating baths. R. SCHNEIDEWIND, S. F. URBAN, and R. C. ADAMS, JUN. (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 9 pp.).—From experiments on the electrolysis of a bath containing 234 g./litre of chromic acid and 4.3 g./litre of potassium sulphate it is shown that the current efficiency of chromium deposition is a linear function of the logarithm of the current density. The position of this line and, to some extent, its slope are affected by the temperature, an increase of temperature lowering the current efficiency at given current density. Bright deposits of chromium up to 0.0007 mm. thick on copper were obtained when the relation between current density and temperature was such as to give

a current efficiency between 5 and 20%, but the optimum current efficiency, which permits of the production of heavy deposits, is about 13%. A diagram shows the current density required to produce any given current efficiency at any given temperature. The relation between current density and current efficiency at 25° is shown to be quite unaffected by the presence of considerable amounts of trivalent chromium and iron in the bath, but the range of current densities within which bright deposits are obtained is notably reduced thereby. It is believed that trivalent chromium is mainly responsible for this effect (cf. Schneidewind and Urban, preceding).

H. J. T. ELLINGHAM.

Hardness and structure of deposited chromium. L. E. and L. F. GRANT (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 11 pp.).—Chromium was deposited from a solution containing 209 g. of chromic acid, 23 g. of chromic oxide, and 6.4 g. of chromic sulphate per litre on steel specimens which had been polished, brass plated, buffed, and then nickel plated. The hardest deposits, according to tests with the Bierbaum apparatus, were obtained at relatively high current densities and the softest at low current densities. At about 20 amp./dm.² good deposits were obtainable over the temperature range 30–60°; the deposit produced at 45° was harder than those at 30° or 60°. The hardest deposits obtained were produced at 45° and 33.3 amp./dm.² The occurrence of a network of fine cracks was observed in some of the deposits, and in some cases a series of networks could be seen underlying the surface network. Preliminary observations on specimens exposed to corrosive conditions suggest that corrosion of the underlying metal occurs more readily along the lines of the network.

H. J. T. ELLINGHAM.

Photomicrographic study of rough or nodulised electrodeposited nickel. C. UPTHEGROVE and E. M. BAKER (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 24 pp.).—A carefully cleaned steel surface was plated successively with about 0.01 mm. of flash nickel, 0.10 mm. of copper, and 0.02 mm. of nickel. Nodules developed on a particular specimen were examined and numerous photomicrographs of horizontal and vertical sections through the nodules are reproduced. The nodules appear always to be the result of outgrowths originating in the normal flash nickel deposit, and the extent of the outgrowths determines the size of the nodule. In no case could the presence of the nodules be related to defects or irregularities of the steel surface or to the method of cleaning, for nodules may start to develop after some thickness of nickel has been deposited. But the flash nickel outgrowths were invariably of a dirty or porous character, and it is believed that in general the nodules are caused by suspended particles of impurities in the bath coming into contact with the metal surface after deposition has started. Hence the use of clear solutions is important for the avoidance of nodular deposits.

H. J. T. ELLINGHAM.

Electroplating on aluminium and its alloys. H. K. WORK (Amer. Electrochem. Soc., April, 1928, 53.

Advance copy. 24 pp.).—An extensive investigation has been made into the conditions under which commercial aluminium and its alloys can be successfully electroplated. For plating on relatively smooth aluminium surfaces, deposition of zinc from a cyanide bath containing free ammonia proved most satisfactory. Nickel, copper, cadmium, or brass can then be deposited over this initial layer of zinc. The plating so produced adheres well, but fails rapidly in corrosion tests, so that it is only satisfactory for dry, indoor service. Improved resistance to corrosion can be attained by heat-treatment of aluminium which has been plated with zinc followed by copper. After heat-treatment the surface may be buffed and nickel plated. Travers' process (B., 1918, 272 A), in which aluminium is plated directly with nickel and then heat-treated at relatively low temperatures for a long time was also tested. As a result of these tests and of trials of other processes described in the literature, it is concluded that plating on smooth aluminium is of doubtful value unless followed by heat-treatment, whilst processes involving heat-treatment are subject to several objections in other ways. Attention was, therefore, given mainly to methods of plating on a surface of aluminium or of its alloys which had been roughened by treatment with a suitable etching solution. It is found that the solutions most suitable for preliminary degreasing of the metal surface and for subsequent etching vary with the chemical and physical character of the alloy. The compositions of such solutions and the period of dipping suitable for various commercial alloys are tabulated. When the surface has been etched, nickel appears to be the most satisfactory metal for the initial plating, and nickel baths of very varied types are suitable for this purpose. The adhesion and corrosion-resistance of electrodeposited coatings 0.013 mm. thick on commercial aluminium and various alloys are tabulated, and photomicrographs showing the mode of adherence of the plating to the etched surface are given. H. J. T. ELLINGHAM.

Electrodeposition of thallium. O. W. BROWN and (Sister) A. MCGLYNN (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 9 pp.).—The electrolysis of baths containing thallium sulphate, fluosilicate, or perchlorate was investigated and the perchlorate type shown to be most promising. Conditions for the deposition of thallium from perchlorate baths were then examined in detail. Good deposits were obtained at room temperature from solutions containing 40–120 g./litre of thallous perchlorate with 10 g./litre of free perchloric acid, 10 g./litre of peptone, and 5 c.c./litre of cresylic acid at 0.4 amp./dm.² Addition of cresylic acid gives smooth, instead of spongy, crystalline deposits, and peptone prevents the formation of thallic oxide on the thallium anode. Electrolysis at a higher temperature (50–60°) improves the deposit, and current densities up to 1.8 amp./dm.² can then be used with concentrated perchlorate solutions containing as much as 60 g./litre of free perchloric acid. H. J. T. ELLINGHAM.

Metallurgical cokes. BRAUNHOLTZ and BRISCOE. **Sardinian fuels.** BINAGHI and BRUNDO.—See II. **Aluminium electrolytic condensers.** SIEGMUND.—See XI.

PATENTS.

Ore concentrating machines. J. R. BROADLEY (B.P. 289,178, 28.1.27).—In concentrating machines of the Wilfley type, the table is given a controlled longitudinal motion by means of an attached yoke driven by toggle levers, cams, etc. against the resistance of springs.

C. A. KING.

Precious-metal recovery machine. C. LOVERIDGE and A. E. COX (U.S.P. 1,668,162, 1.5.28. Appl., 6.10.25).—The machine has flat reciprocating screens arranged with jump-gaps, and material passing through the latter falls upon a stratifying table placed below. Material passing over the gaps is discharged.

F. G. CLARKE.

Flotation of ores. G. G. GRISWOLD, JUN., and G. E. SHERIDAN (U.S.P. 1,668,202, 1.5.28. Appl. 6.3.24).—Prior to being subjected to a froth flotation process, an ore containing sulphides of lead, copper, zinc, and iron is treated with sufficient chlorine to inhibit the subsequent flotation of the sulphides of zinc and iron.

F. G. CLARKE.

Mechanical [ore-roasting] kilns. ERZRÜSTUNG GES.M.B.H., and G. BALZ (B.P. 288,823, 25.4.27).—In a kiln of the multiple-shelf type the stirring vanes are attached to a carrier arm of relatively light construction which is supported at both ends. The arms may be rotated by mechanical means operating either on the central shaft or on supporting rings at the peripheral ends of the arms.

C. A. KING.

Reduction of metallic oxides. W. H. SMITH (B.P. 266,289, 20.10.26. U.S., 17.2.26).—A mixture of iron ore or other metallic oxide and a reducing agent is caused to pass continuously through horizontal, closed, rectangular retorts arranged in banks. The retorts are heated to about 1100° by means of separate heating flues arranged between the retort benches. Heat is recuperated from the cooling metal.

C. A. KING.

Preparation of pig iron in highly-heated mixers. K. STOBRAWA (U.S.P. 1,666,428, 17.4.28. Appl., 22.1.27. Ger., 16.2.26).—Pig iron rich in carbon is pre-heated on the flat hearth of a highly-heated mixer by the addition of scrap iron with a low carbon content which has been heated nearly to its m.p. in a separate furnace. The scrap iron is immediately carbonised and its m.p. reduced, while the carbon, silicon, and phosphorus content of the pig iron is reduced without substantially reducing the manganese content.

A. R. POWELL.

Manufacture of steel. KABUSHIKI KAISHA NIHON SEIKOSHO (B.P. 273,248, 12.3.27. Japan, 22.6.26).—In the heat-treatment of steel to remove internal stresses and to increase toughness, the steel is quenched from a temperature above the Ac₃ point, reheated to rather above the Ac₁ point, and slowly cooled to rather below the Ar₁ point. The metal is then annealed between Ac₁ and Ar₁ and slowly cooled.

C. A. KING.

Ferrous alloys. B. D. SAKLATWALLA (B.P. 288,861, 30.6.27).—An alloy containing 1–8% Cr, 0.5–5% Cu, 1–6% Si, with or without carbon up to 3.5%.

C. A. KING.

Steel alloys. F. KRUPP A.-G. (B.P. 276,317, 25.7.27. Ger., 23.8.26).—Heat-resisting steel alloys

contain 15–25% Cr, 15–25% Ni, and less than 0.2% C. C. A. KING.

Coating of metallic articles to protect them from corrosion. R. NOVELLY (B.P. 288,674, 1.1.27).—The metal sheets are provided on one side or on both sides with a thin sheet of an alloy of iron with 6–25% Cr, 20–0.5% Ni, and up to 1% C. A. R. POWELL.

Purification of antimonial lead alloys. H. WADE. FROM U.S. SMELTING, REFINING, & MINING Co. (B.P. 288,939, 21.12.27. Addn. to B.P. 236,782; B., 1925, 727).—Arsenic may be removed from antimonial lead or lead-tin alloys by blowing the molten alloy with a current of air under a flux of molten sodium hydroxide at 335–480° (preferably 450–460°). A. R. POWELL.

Manufacture of alloys. W. M. GROSVENOR and V. P. GERSHON (U.S.P. 1,168,642, 8.5.28. Appl., 3.2.25).—An alloy, having the colour of platinum and capable of being cast or rolled for jewellery, is obtained by melting a base-metal alloy (about 80% Ni and 20% Zn) with 3 pts. by wt. of fine gold. F. G. CROSSE.

Manufacture of metal catalysts. I. G. FARBERIND. A.-G. (B.P. 282,410, 14.12.27. Ger., 14.12.26. Addn. to B.P. 281,218; B., 1928, 235).—Metallic catalysts such as nickel, cobalt, and copper, or mixtures thereof, may be made by precipitation from their ammoniacal solutions by the action of hydrogen or a gas containing hydrogen, preferably under pressure, in the presence or absence of a carrier. The precipitation may be accelerated by the presence on the carrier of a metal having a catalytic action such as nickel, cobalt, or copper.

M. E. NOTTAGE.

Bimetallic [thermostatic] element. H. SCOTT, ASSR. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,665,935, 10.4.28. Appl., 27.12.26).—The elements, having different temperature coefficients of expansion, consist of a ferrous alloy containing 42% Ni and of a ferrous alloy containing nickel and 1% Mn.

A. R. POWELL.

Electrolytic extraction of tin from alkaline lyes. SIEMENS & HALSKE A.-G. (B.P. 286,673, 7.3.28. Ger., 8.3.27).—In order that the tin may be removed in a compact form, the electrolyte or the initial material, before electrolysis, is treated with an additional and relatively powerful oxidising agent such as ozone, hydrogen peroxide, or the peroxides of the alkali or alkaline-earth metals, until practically all the tin is present in the form of stannate; a cathodic-attractive colloid such as gelatin is then added.

M. E. NOTTAGE.

Electrolytic recovery of pure tin from impure stanniferous materials. BERZELIUS METALLHÜTTEN GES.M.B.H. (B.P. 271,521, 23.5.27. Ger., 21.5.26).—Tin may be recovered from tin alloys containing less than 80% Sn by electrolysis of the alloy in the form of fine shavings or granules produced by pouring the molten alloy at a high speed into a quenching liquid. The particles should preferably have a thickness not exceeding 2 mm. and are loosely held together in a permeable container which forms the anode in a sodium hydroxide electrolyte.

A. R. POWELL.

Method and alloy for galvanising metal articles. J. H. LAWES (B.P. 288,741, 21.1.27).—Instead of using

chloride fluxes above the galvanising bath, the bath itself is treated from time to time with small quantities of an alloy of 1 pt. of aluminium with 40 pts. of zinc.

A. R. POWELL.

Apparatus for the spraying of molten substances [metals]. C. J. JUNG (B.P. 276,955–6, 29.4.27. Holl., 3.9.26).

Production of [catalytic] platinum contact bodies. T. VON ARTNER (B.P. 265,938, 18.1.27. Ger., 15.2.26).—See G.P. 440,338; B., 1927, 813.

Briquetting of ores (B.P. 274,876).—See II. **Enamels for metals** (B.P. 273,260).—See VIII.

XI.—ELECTROTECHNICS.

Caustic soda primary battery. M. L. MARTUS (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 17 pp.).—The construction of primary cells of the Lalande type used by the American railroads on signal and track service is described. The copper-copper oxide electrode (cathode) is either of the “loose element type” consisting of powder, granules, or flakes of cupric oxide in a perforated or porous container, or of the “compressed oxide element type” prepared by mixing finely-ground cupric oxide with a binder, such as a solution of sodium or potassium hydroxide or copper sulphate, placing in forms of various shapes, compressing, baking, and treating to produce partial superficial reduction. The latter type is now more widely used, and the electrode is generally in the form of a flat plate or a hollow cylinder. The importance of setting up these cathodes and the zinc anodes in the upper part of the electrolyte is indicated by capacity data quoted. Results are given showing the effect of temperature and the electrolyte concentration on the capacity of the battery expressed as the number of amp.-hrs. obtained during discharge at 3 amp. rate until the voltage fell to 0.5. The higher the concentration of sodium hydroxide, within certain limits, the more efficient is the cell at high temperatures and the less efficient at low temperatures. The variation of capacity with temperature is least when the concentration of the electrolyte is about the standard value, 1050 g. of sodium hydroxide in 4400 c.c. of water. The cells are manufactured commercially in sizes from 75 to 1000 amp.-hr. capacity.

H. J. T. ELLINGHAM.

Adsorption phenomena in primary cells. E. O. JEGGE (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 8 pp.).—Capacity for adsorbing ammonia from solution is an important property of manganese dioxide for use in the manufacture of dry cells. Thus, pyrolusite from Montana, although it contains only 70–72% MnO₂ is now preferred to the richer Caucasian and Javanese ores, since, owing to its greater adsorptive power for ammonia, it keeps down the alkalinity of the electrolyte and thus lowers the polarisation of the cell during discharge. On the other hand a cell of the Leclanché type can be set up in which charcoal pressed around a carbon rod constitutes the cathode system and no manganese dioxide is used. With such a cell containing 600 c.c. of a saturated solution of ammonium chloride as electrolyte, a zinc anode, and a cathode weighing about 100 g., discharging through

10 ohms down to 0.9 volt gave 45—50 amp.-hrs. After renewing the electrolyte and, if necessary, the zinc, a further discharge can be obtained, and by repeated renewals of electrolyte and zinc and repeated discharges down to 0.8 volt as much as 400 amp.-hrs. may be obtained before the pores of the charcoal become clogged with salts. The theory of the action of the cell is discussed with reference to data for the adsorptive capacity of the charcoal.

H. J. T. ELLINGHAM.

Drying and storage of secondary battery plates in the charged state. I. Characteristics of plates dried in carbon dioxide in the charged condition. S. MAKIO (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 6 pp.).—Positive and negative plates were fully charged, washed, and dried in carbon dioxide, and then wrapped in paper and stored for 6 months. On setting up batteries with these plates in sulphuric acid (*d* 1.20), a constant terminal voltage somewhat over 2 volts was attained in about 1 hr., and the capacity of the battery, calculated from the first discharge, has an average value of 79% of the rated amount.

H. J. T. ELLINGHAM.

Theory of the lead storage battery. L. JUMAU (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 6 pp.).—The theory put forward by Féry (cf. B., 1925, 997) as to the chemical charges occurring during the operation of the lead accumulator is discussed critically. Féry's own experimental results on the change in weight of the positive plate during discharge are corrected for change in buoyancy due to change in volume of the active material, and are then found to agree closely with Gladstone and Tribe's classical double-sulphation theory and not at all with Féry's theory. Moreover, Féry's assumption that plumbous sulphate is formed at the negative plate has no sound basis. It is concluded that the double-sulphation theory accounts fully for the reactions occurring in the lead accumulator.

H. J. T. ELLINGHAM.

Physico-chemical aspects of the Leclanché dry cell. F. DANIELS (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 20 pp.).—The reactions occurring during the discharge of a Leclanché dry cell are discussed. Measurements of single potential were made on electrodes of manganese dioxide electro-deposited on platinum. The potential was found to depend on the p_{H} value of the solution according to the relation $e = e' - 0.075p_{\text{H}}$ over the p_{H} range 5—10, where e' is a constant, but to be independent of whether ammonia and ammonium chloride or citric acid and disodium phosphate buffer mixtures were used as the electrolyte. From these data and other evidence the suggestion is made that the fundamental depolarisation reaction in the Leclanché cell is $[\text{Mn}(\text{OH})_3] + \ominus = [\text{Mn}(\text{OH})_2] + \text{OH}'$, the ions of the former type being produced by hydrolysis of manganese dioxide. With manganese dioxide electrodes which had been partially reduced by heating or cathodic treatment, extremely base values of the single potential were obtained at first, but they soon became more positive and eventually approached the normal value corresponding with the p_{H} value of the solution. The base values are attributed to the presence of manganous hydroxide, and it is

believed that this substance may be produced when very heavy currents are taken from a dry cell or when a cell has run down. The conditions for the precipitation of $\text{ZnCl}_2 \cdot 2\text{NH}_3$ are examined. After much electricity has been taken from the cell, so that the ammonium chloride concentration has fallen considerably, precipitation of $\text{OH} \cdot \text{ZnCl}$ may also occur. The factors determining the voltage relations during discharge under various conditions are discussed and the question of shelf deterioration is considered.

H. J. T. ELLINGHAM.

Furfural[dehyde] as a possible ionising medium. J. R. MCKEE, C. A. MANN, and G. H. MONTILLON (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 17 pp.).—The electrical conductivity of various inorganic salts dissolved in furfuraldehyde, stabilised by the addition of 0.1% of pyrogallol, was determined. Only the halides, and especially the iodides, are soluble to any great extent in furfuraldehyde, and solutions of ammonium iodide, potassium iodide, zinc iodide, ammonium bromide, ammonium chloride, and ferric chloride had appreciable conductivities. The conductivity of furfuraldehyde stirred with ferrous, chromic, or cuprous iodides was unchanged, but cuprous and zinc iodides are good conductors in solutions of ammonium iodide in furfuraldehyde. Cuprous iodide dissolves more readily in furfuraldehyde solutions than in aqueous solutions of ammonium or potassium iodide, and the solubility and conductivity of cuprous iodide in furfuraldehyde solutions of the ammonium halides increases with the mol. wt. of the halide. Experiments were made to determine whether metals could be electrodeposited from furfuraldehyde solutions of their salts. Electrolysis of a solution of cuprous and ammonium iodides in furfuraldehyde at about 0.5 amp./dm.² with a copper anode and platinum cathode gave a copper deposit with a current efficiency of 56.5%. From a solution of silver and potassium iodides in furfuraldehyde, silver was deposited as a loose, granular deposit at very high current efficiency. But attempts to deposit zinc from iodide solutions were unsuccessful owing to the formation of tar on the electrodes and to notable reduction of the furfuraldehyde. Electrolytic corrosion of a tungsten anode in a furfuraldehyde solution of ammonium iodide did not occur, and the furfuraldehyde was changed to a rubbery tar.

H. J. T. ELLINGHAM.

Aluminium electrolytic condenser. H. O. SIEGMUND (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 25 pp.).—The electrical properties of aluminium anode films, the sources of energy loss in aluminium condensers, and the conditions affecting their life are discussed and the construction of the commercial form of the apparatus is described and illustrated. The positive electrodes consist of corrugated aluminium plates. The higher the purity of the aluminium, the more rapid is the formation of the film and the lower the resultant leakage current, but failures due to corrosion are decreased by the use of aluminium containing a notable proportion of silicon. Data for the specific resistance of solutions containing ammonia and boric acid in various proportions are given, and the effect of electrolyte composition on the performance and life

of the condenser is referred to. Curves showing the effect of corrosion of the anodes on capacity and leakage current are recorded, and the variation of the capacity of the condenser during operation over a period of several years is also illustrated. H. J. T. ELLINGHAM.

Photoelectric turbidimeter. L. W. HAASE and H. THIELE (Gas- u. Wasserfach, 1928, 71, 414—417).—The turbidities of liquids have been satisfactorily determined relative to distilled water by examining them in a cylindrical cell closed at one end by a parallel-sided glass plate illuminated with constant energy, and at the other by a selenium cell. This selenium cell is connected to form one arm of a Wheatstone bridge, and its *E.M.F.* is balanced in the usual way against a constant *E.M.F.* from an accumulator. A. E. MITCHELL.

Attack of cotton by oils. STÄGER.—See V. **Distillation of acids.** HOFF.—See VII. **Melting silver.** LEACH. **Chromium plating baths.** SCHNEIDEWIND and others. **Chromium.** GRANT and GRANT. **Nickel.** UPTHEGROVE and BAKER. **Plating on aluminium and its alloys.** WORK. **Thallium.** BROWN and MCGLYNN.—See X. **Cane juice defecation.** BALCH and PAINE.—See XVII.

PATENTS.

High-frequency electric induction furnace. R. DUFOUR (B.P. 275,611, 28.7.27. Belg., 4.8.26).—An inductor element, separate from and not attached to the furnace walls, projects into the heating chamber through an opening in the base. J. S. G. THOMAS.

Electrodes [for electric arcs]. W. IRBY, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,669,129, 8.5.28. Appl., 7.1.27).—The electrode consists of ilmenite, chromite, magnetite, sodium fluoride, and lithium fluoride.

F. G. CROSSE.

Activation of electron-emission material [tungsten filaments]. J. W. MARDEN and J. E. CONLEY, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,665,636, 10.4.28. Appl., 21.2.23).—Tungsten powder is mixed with a small proportion of tungsten carbide, and the mixture pressed into rods which are sintered, swaged, and drawn into filaments. A. R. POWELL.

Electromagnetic bodies. SIEMENS & HALSKE A.-G. (B.P. 271,106, 13.5.27. Ger., 17.5.26).—Ferromagnetic monocrystalline substances, e.g., iron, produced by the decomposition of iron carbonyl at a temperature between 100° and 500° under a pressure less than atmospheric, and possessing practically negligible hysteresis losses, are used as cores for electromagnetic relays, transformers, etc. J. S. G. THOMAS.

X-Ray tube. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,665,637, 10.4.28. Appl., 18.6.27).—An X-ray tube having a molybdenum cathode and a uranium anode is claimed. Uranium has better ray-emitting properties than tungsten. A. R. POWELL.

Insulating composition (B.P. 280,178).—See V. **Recovery of tin** (B.P. 271,521).—See X.

XII.—FATS; OILS; WAXES.

Composition of fatty acids of palm oil. A. RAYNER and S. G. CAMPBELL (J.S.C.I., 1928, 47, 149—150 r).—The general properties of several grades of

palm oil are recorded including the contents and properties of the solid acids. It is shown that (1) the titre and iodine value and solid acid content of palm oils may vary to a considerable extent according to the source of supply of the oil; (2) the difference in these characteristics is due to variations in the amount of solid acids present, and not to any great variation in the composition of the latter; (3) the proportion of stearic acid in the solids is considerable, and varies from 12.5% to 18.5%; (4) the liquid acids calculated from the iodine value contain, on an average, 20.0% of linoleic and 80.0% of oleic acid.

Rational selection of soapmaking properties by means of a new chemical control. S. PRAGER (Chem. Umschau., 1927, 34, 327—330; cf. E. T. Webb, "Modern Soap and Glycerin Manufacture").—By means of the *INS* factor (iodine value — saponif. value) and *SR* (solubility ratio) it is possible to control the hardness, solubility, and lathering properties of soap. For linseed oil the *INS* factor is 15, for palm kernel oil 235, and for coconut oil 250. The relative hardness which an oil imparts to a soap is measured by the *INS* factor, this value varying directly with the hardness. Suitable blending of soft and hard oils enables a soap of any desired hardness to be made (preferably with *INS* factor of 150 for the mixed fats), and simple numerical equations afford a means of calculating the percentage of each oil necessary to give any desired *INS* factor in the mixture. The solubility ratio is determined by dividing the *INS* factor ($\times 100$) of the mixed fats by the sum of all the *INS* factors above 130 (excluding palm kernel and coconut oils) multiplied by the percentages of the respective constituents. For *INS* factors between 130 and 160 the *SR* value should not be below 1.50, though it may be above 2. The solubility ratio is a measure of the solubility and the lathering power of soap: the higher this value the greater are these properties. H. M. LANGTON.

Transformation of fats into hydrocarbons. B. MELIS (Annali Chim. Appl., 1928, 18, 108—114).—Results are given of the fractional distillation of the hydrocarbon oil obtained when a mixture of the sodium and calcium salts prepared from animal and vegetable fats is subjected to dry distillation (B., 1925, 235). The percentage composition of the oil is: b.p. below 150° d_0 0.706—0.759, 20.99; b.p. 150—310°, d_0 0.786—0.843, 45.33; b.p. above 310°, d_0 0.860, 22.66; tar, 7.00; loss, 4.02. These numbers correspond closely with those furnished by the oil obtained by dry distillation of fats in presence of zinc chloride (cf. Mailhe, B., 1925, 534), and differ from those given when natural petrol- eums are distilled mainly in the higher proportion of light oils. The oil contains aromatic and terpenic products and, indeed, members of all the series represented in natural petrol- eums; it would probably correspond more closely with the latter if the dry distillation were carried out under pressure, so as to favour rehydro- generation. T. H. POPE.

Determination of iodine values by means of aqueous solutions. A. HANSEN (Dansk Tidsskr. Farm., 1928, 2, 89—133).—Winkler's method for the determination of iodine values (cf. B., 1916, 305) has been critically

examined. The addition of the halogen proceeds rapidly at first, but the rate diminishes greatly after about 80–90% of the total halogen has entered into combination, and shaking the reaction mixture does not appear to accelerate the reaction. Losses due to the volatility of bromine can be prevented by employing 5 g. of potassium bromide instead of the 1 g. originally proposed by Winkler. The mixture with 1 g. of bromide gives a somewhat more rapid addition, of halogen, and yields results which are slightly higher than those obtained by using 5 g., but both mixtures give iodine values which are in good agreement with those obtained by Hübl's method. Using 5 g. of potassium bromide the reaction is complete in 2 hrs. in the case of non-drying oils, but requires 20 hrs. in determinations of drying or partially-drying oils. Prolongation of the time of reaction beyond these limits has no appreciable effect on the result. The iodine values obtained are influenced by the excess of halogen present in the reaction, the best values being given when an excess of 40–50% over and above the theoretical amount is present. It is consequently recommended to use an amount of the substance under examination corresponding to the formula weight w (grams) = $33 \div$ presumed iodine value of substance. Winkler's solutions exposed to a light of 600 c.p. in a special apparatus for varying periods at 20° showed no liberation of bromine, but a series of similar experiments with the addition of olive oil gave irregular results for the iodine value, varying from 90.25 to 102.6. This is ascribed to the reaction taking place in two stages consisting first in the addition of halogen, and then of substitution. During the first period the mixture is not sensitive to light, but becomes so during the later stage, mixtures containing 1 g. of potassium bromide being more sensitive than those containing 5 g. The use of 5 c.c. of ether with 5 c.c. of carbon tetrachloride as a solvent, and 25 c.c. of a 0.2*N*-solution of potassium bromate containing 0.415 g. of potassium iodide and 0.3 g. of potassium bromide as reaction mixture diminishes the sensitivity of the process to light, and shortens the time required for the reaction, but cannot be recommended, as low results are obtained unless the excess of halogen amounts to at least 40%. The following modification of Winkler's original method is finally recommended as the most satisfactory:—A quantity of the substance in grams equal to $33 \div$ presumed iodine value is weighed out into a flask fitted with a ground-in glass stopper, and dissolved in 10 c.c. of pure carbon tetrachloride, 5 g. of potassium bromide in 15 c.c. of water and 25 c.c. of 0.2*N*-potassium bromate solution are added, followed by 10 c.c. of 10% hydrochloric acid. The stopper is quickly inserted and the flask rotated several times to cause the bromine to dissolve in the tetrachloride. The flask is then placed in a metal canister, painted black internally and externally, and rendered light-tight, and left there for from 2 to 20 hrs. according to the nature of the substance under examination. The covering and stopper are then removed and, without taking the flask out of the canister, 10 c.c. of 10% potassium iodide solution are poured in, and, after an interval of a few minutes, 150 c.c. of water are added. The liberated iodine is then titrated with 0.1*N*-thio-sulphate, the liquid being shaken well during the

titration so as to ensure complete decolorisation of the tetrachloride layer. H. F. HARWOOD.

Detection of refined olive oil in virgin olive oils. A. BAUD and COURTOIS (*Chim. et Ind.*, 1928, 19, 602–603).—See B., 1928, 201.

Viscosity of oils. RAASCHOU.—See I. **Determination of glycerin.** KELLNER.—See III. **Wax from sugar cane.** BARDORF.—See XVII.

PATENTS.

Extraction of oils and the like from bodies containing the same. L. J. SIMON, and SIMON EXTRACTING MACHINE SYND., LTD. (B.P. 288,670, 14.12.26. Cf. B.P. 255,923; B., 1927, 49).—Oils, fats, and waxes are extracted by subjecting the material to repeated flushings with solvent while disposed in a basket rotating in a closed extraction vessel from which the solution is withdrawn and re-introduced into the centre during each flushing. This process provides a more expeditious method of manipulating the solvent and solutions from the various flushings. E. HOLMES.

Production of oils containing vitamins. BRIT. DRUG HOUSES, LTD., and F. H. CARR (B.P. 289,187, 7.2.27).—A solution of vitamins suitable for pharmaceutical purposes, or for use in food products, may be obtained by extracting mammalian livers with vegetable, animal, or mineral oils, with or without raising the temperature to 55–60°. E. HOLMES.

Soap powder and the like. LEVER BROS., LTD., and R. THOMAS (B.P. 288,654, 4.10.26).—A washing and bleaching agent capable of exerting its bleaching action in the presence of water at ordinary temperatures may be prepared by incorporating with soap flakes, powder, or bars, varying amounts of a double compound of trisodium phosphate and sodium hypochlorite, with or without sodium carbonate and other usual soap-powder ingredients. If kept substantially dry the double salt is stable at least for several months even in the presence of the organic matter of the soap. E. HOLMES.

Manufacture of rosin soap. C. C. KESLER, ASSR. to PINE INSTITUTE OF AMERICA, INC. (U.S.P. 1,663,764, 27.3.28. Appl., 30.7.25).—The resene component of the rosin is removed after the acid component has been converted into an alkali hydrogen salt, and the salt is used in the manufacture of soap. L. A. COLES.

Production of aqueous solutions of organic compounds insoluble or difficultly soluble in water. I. G. FARBENIND. A.-G. (B.P. 261,720, 7.10.26. Ger., 19.11.25).—Aqueous solutions of sulphonic acids having soap-like properties (especially aralkylated sulphonic acids, *e.g.*, the isopropylated acids of G.P. 336,558; B., 1921, 630 A) or their salts have their property of dissolving insoluble organic compounds, *e.g.*, oils, fats, hydrocarbons, etc., considerably increased by the addition of soluble salts, such as Glauber salt. [Stat. ref.] B. FULLMAN.

Production of sulphuric esters of poly[hydr]oxy-fatty acids. H. FLESCH (B.P. 287,076, 4.8.27. Ger., 14.3.27).—The laboratory preparation of dihydroxystearic acid (*cf.* Grün, B., 1907, 57) can be adapted to the production of sulphuric esters of dihydroxy-

trihydroxy-, and polyhydroxy-fatty acids on the technical scale by treating the cooled and actively agitated hydroxyfatty acid with sulphuric acid in the form of a very fine spray, and with sulphuric acid containing 5–10% of sulphuric anhydride. E. HOLMES.

Production of sulphonated oils and fats with a high content of organically combined sulphuric acid. H. FLESCH (B.P. 282,626, 4.8.27. Ger., 23.12.26).—The interaction of unsaturated oils, fats, and fatty esters, and glacial acetic acid with sulphur trioxide or chlorosulphonic acid gives high-grade sulphonates which have a higher content of organically combined sulphuric acid and a higher wetting power than Turkey-red oils. E. HOLMES.

Fluid composition [metallic soap] applicable as a covering, colouring, or dispersing agent, etc. C. J. J. Fox (B.P. 289,122, 15.1.27).—A dispersion medium consisting essentially of a strong ammonium base and the soap of a fatty acid or mixture with metals such as zinc, copper, or nickel, which give ammonia-soluble hydroxides, but which are insoluble in water alone, may form the basis of numerous technically useful products. Such a dispersion may, for example, be emulsified with pigments, insect poisons, or rubber latex to give sheep-marking fluids, insecticides, and water-proofing liquids, respectively. Such products are water-resistant after the base has evaporated, yet may be readily removed with dilute ammonia, ammonium chloride, or sodium carbonate. E. HOLMES.

Recovery of fatty material from emulsions. Soc. FRANÇ. DE CENTRIFUGATION, and J. A. L. ROZIERES (B.P. 289,350, 25.10.27).—In an apparatus for the extraction of fatty materials from such emulsions as wool scourings, by means of a counter-current of a volatile solvent which is subsequently removed by centrifuging, special provision is made to prevent clogging of the apparatus by solid matter, as pulverised wool. E. HOLMES.

Bleaching liquors (B.P. 289,156).—See VI.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Colloid chemistry of paints, varnishes, and their components. R. S. MORRELL and W. E. WORNUM (J. Oil and Colour Chem. Assoc., 1928, 11, 49–62, 93–112).—A digest of the literature on the colloid chemistry of paints, drying oils, resins, and varnishes, covering the period 1920–1927 (cf. "Third Report on Colloid Chemistry and its General and Industrial Applications," Dept. Sci. Ind. Research, 1920).

S. S. WOOLF.

The red lead question. [Action of red lead in rust-protecting paints.] H. WOLFF and R. SINGER (Farben-Ztg., 1928, 33, 1909–1913).—Rahder's statement (B., 1928, 99) that red lead accelerates the corrosion of iron is true only for the system red lead–iron–water, and does not hold for paints in which red lead forms the pigment. The oil adsorbed by highly dispersed red lead may vary very considerably, and is of more importance in determining the rust-protecting capacity of a paint than is the total oil content. The oil content, degree of dispersion of the pigment, and the

swelling capacity of a paint are not interdependent functions. The last-named depends on the free oil content, i.e., on the oil which is not adsorbed on the surfaces of the particles of pigment. The protective action of the paint is due to a thin, almost molecular film in which the oil is present as an extraordinarily strong adsorption complex with the pigment and the base. The swelling capacity of the upper layers cannot therefore be a measure of the protective action of the paint, which is much more likely to be due to the formation of a protective film on the surface of the iron.

A. R. POWELL.

Determination of iron in red lead. H. HEINRICHS (Z. angew. Chem., 1928, 41, 450–453).—The usual method of determining iron in red lead by dissolving the sample in nitric acid, precipitating the lead as sulphate, and weighing the iron as oxide gives low results owing to the insolubility of part of the iron in nitric acid and to adsorption of ferric sulphate by the lead sulphate precipitate. Correct results may be obtained by the following colorimetric method: 10 g. of the sample are mixed with 25 c.c. of a cold saturated solution of hydroxylamine hydrochloride, and the mixture is boiled for 10 min. with 50 c.c. of hydrochloric acid (d 1.19). The lead chloride is dissolved completely in 400 c.c. of boiling water and the solution set aside overnight. The liquid is decanted, evaporated to 100 c.c., and again allowed to crystallise. The second filtrate is treated with ammonia, the ferric hydroxide precipitate collected and dissolved in hydrochloric acid, and the iron determined colorimetrically with thiocyanate. A blank test should be carried out using the same quantities of reagents as in the analysis. A small, grey, spongy residue after dissolving the sample in the acid mixture is metallic lead, the presence of which is objectionable in red lead used for glass frits.

A. R. POWELL.

Bleaching of lac. M. VENUGOPALAN (J. Indian Inst. Sci., 1928, 11A, 17–22).—Lac may be bleached by dissolving it in 2.5% sodium carbonate solution at 60–70° and, after filtration and cooling to air temperature, adding a solution prepared by passing chlorine into a solution containing 12.5% of caustic soda and 2.5% of sodium carbonate. The latter should contain 6–8% of available chlorine and, if of p_H 10–10.5, does not require storing in a cool place. The amount of such a solution necessary for bleaching indicates a chlorine requirement of 10–14% on the weight of lac, and a yield of 93–95% is obtained. The bleached lac may be recovered by the slow addition, with stirring, of 1:20 sulphuric acid, the precipitate being then collected, washed, and dried *in vacuo* over sulphuric acid. The product is freely soluble in cold 97% alcohol, and the solubility does not alter on prolonged storage in air. The bleached material contained 2.3–3.1% of moisture, 0.98–3.52% Cl, and had saponification value 236.0–256.7, acid value 70.68–83.52, and iodine value 3.9–5.0.

F. E. DAY.

PATENTS.

Manufacture of white oil paints from lithopone and manufacture of lithopone. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 289,153, 21.1.27).—Thickening of white oil paints manufactured from litho-

pone is hindered by the presence of at least sufficient free fatty acids to prevent or minimise the thickening action of the zinc oxide present. Linseed oil may be used containing a sufficiently high proportion of the fatty acids, or these may be added in colloidal suspension or dispersion to the lithopone. L. A. COLES.

Steam turpentine still. J. O. REED, Assr. to U.S.A. (U.S.P. 1,667,168, 24.4.28. Appl., 28.7.27).—Steam is introduced into the bottom of the still through a vertical supply pipe which communicates with radial jets, and between the supply pipe and the jets is a valve operated by the steam pressure. F. G. CLARKE.

Enamel for coating cans [to contain foodstuffs]. R. H. LUECK, Assr. to U.S.A. (U.S.P. 1,667,212, 24.4.28. Appl., 23.3.26).—A neutral oleoresin varnish is incorporated with compounds of metals forming sulphides which are not dark in colour. L. A. COLES.

Plastic material. Flexible moulded articles from condensation products. E. L. AIKEN, Assr. to CARBOLOID PRODUCTS CORP. (U.S.P. 1,668,444—5, 1.5.28. Appl., 23.9.21. Renewed [B] 25.4.24).—(A) Phenol and glycerol are condensed by a dehydrating agent. (B) The product is condensed with "methylenic," and the final material shaped without pressure and heated. B. FULLMAN.

Synthetic resin. C. E. BURKE and H. H. HOPKINS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,667,189, 24.4.28. Appl., 28.4.27).—A mixture of pentaerythritol, a polybasic acid, and a drying oil acid is heated to a temperature at which reaction takes place.

A. R. POWELL.

Titanium pigment. J. BLUMENFELD, Assr. to COMMERCIAL PIGMENTS CORP. (Re-issue 16,956, 8.5.28, of U.S.P. 1,504,673, 12.8.24).—See B., 1925, 999.

Fluid composition (B.P. 289,122).—See XII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Preparation of butadiene. S. F. BIRCH (Ind. Eng. Chem., 1928, 20, 474).—By bromination of the gases obtained on distillation of 8 gals. of "railway hydrocarbon" (the liquid obtained during the preparation of oil gas), 1250 g. of solid butadiene tetrabromide and 560 g. of the liquid tetrabromide can be obtained. Pure butadiene can be prepared from the solid derivative by Thiele's method (A., 1900, i, 2) with zinc dust and alcohol. Butadiene obtained similarly from the liquid tetrabromide is probably contaminated with traces of high-boiling olefines. E. H. SHARPLES.

Rubber fibre and cellulose fibre. Recognition of the elastic linking. H. FEUCHTER (Kautschuk, 1928, 4, 73—75; cf. B., 1928, 377).—The "shell-aggregation" theory of the structure of rubber is reviewed and its confirmation by the phenomena of "racking" is indicated. The analogy between the structure of rubber and cellulose extends to the persistence of the structural principle after vulcanisation in the case of racked rubber and after nitration or acetylation in the case of cellulose. D. F. TWISS.

Specifications as to composition and selling conditions for rubber goods. W. ESCH (Kautschuk,

1928, 4, 75—83).—A discussion of the value of specifications fixing the composition of rubber articles for various purposes. In addition to unavoidable variation arising from imperfections in the mixing operations and in subsequent chemical analyses, a disadvantage of such specifications is that they fail to ensure the qualities essential to good service, and, indeed, tend to check the development of newer methods of compounding capable of giving better results. Examples are cited.

D. F. TWISS.

XV.—LEATHER; GLUE.

Extractor for vegetable tanning materials. B. SCHWARZBERG (Collegium, 1928, 151—154).—A cylindrical glass vessel is connected from the bottom to another similar glass vessel containing the tanning material. Both stand in the same water-bath. A tube from the second vessel carries the tanning infusion so that it siphons into a litre flask. The first vessel contains 100—200 c.c. of distilled water, which is continually replenished from an inverted litre flask mounted in it and containing distilled water. A rubber connexion and clip in the delivery tube for the extract enables the rate of flow to be regulated. The vessel containing the tanning material can easily, if desired, be fitted up like a Procter extractor, with inverted thistle funnel. The whole apparatus stops automatically in case of choking. D. WOODROFFE.

Gelatins of different origin as emulsifying agents. J. C. KERNOT and J. KNAGGS (J.S.C.I., 1928, 47, 96—100 T).—The contradictory results obtained by different authors while studying the efficiency of gelatins as emulsifying agents are due to the presence of both organic and inorganic impurities, and to the fact that the p_H of the gelatin solutions has not been considered. By working with highly-purified gelatins, it was found that the previous treatment of the precursors has a marked effect on the emulsifying power of the gelatins obtained from them. The viscosity of the gelatin solutions also plays a great part in its emulsifying power. The viscosity also depends on the previous history of the precursor.

PATENT.

Manufacture of [vegetable] glue. P. KREISMANN (U.S.P. 1,667,073, 24.4.28. Appl., 28.3.21).—A mixture of potato starch, clay, water, and an alkali is heated until semi-fluid. H. ROYAL-DAWSON.

XVI.—AGRICULTURE.

Comparison between soil examinations by the seedling method and that of Mitscherlich. H. NEUBAUER, W. BONEWITZ, and A. SCHOTTMÜLLER (Landw. Versuchs-Stat., 1928, 107, 131—142).—Arrangement of a series of soils according to their content of available potash and phosphate as determined by the two methods showed a general parallelism. The average potash values by the seedling method were double, and those for phosphate one half those obtained by the Mitscherlich method. Differences may be partly explained by the difference in the growth period in the two processes. The important difference, however, is that in Mitscherlich's method crop increases result from

added fertilisers which differ in the chemical condition, capability of movement, and distribution in the soil mass from the nutrients present in relatively stable equilibrium in untreated soils as used by Neubauer. The seedling method is not advocated for recently fertilised soils.

A. G. POLLARD.

Effect of dehydration of soils upon their colloid constituents. I—III. J. L. STEENKAMP (Soil Sci., 1928, 25, 163—182, 239—251, 327—332).—I. Dehydration caused a definite decrease in the clay fraction owing to cementing together of small particles. Determinations of the sum of the absorbed bases actually present in the soil, and the degree of saturation in respect to bases at differing stages of dehydration, indicate that on drying an increase in the unsaturated clay acids and in the exchangeable bases present occurs.

II. An increase in acid salt-forming bases takes place on dehydration.

III. Only air-dried soil of high humus content showed reversibility of the colloidal condition after 21 days. The bearing of the above experiments on cultural conditions is discussed.

H. J. G. HINES.

Decomposition of hydrogen peroxide in soils. K. SCHARRER (Landw. Versuchs-Stat., 1928, 107, 143—187).—No definite relationship exists between the catalase activity of soils and their reaction, although, in general, acid soils are less active than neutral or alkaline ones. The catalytic power of soils increases with the amount of iron and manganese compounds present. Chalky soils are very active, and a general relationship exists between catalase activity and the amount of calcium extracted with ammonium chloride solution. Sandy soils are usually more active than loams and clays, and humus soils than mineral soils of similar composition and reaction. Catalytic power in soils is more closely associated with dispersed clay than with the larger soil particles, although these have a definite influence. Mineral salts affect catalase activity according to their ability to flocculate or deflocculate the clay. Boiling or moderate heating of soil reduces its activity and further reduction occurs after ignition. Catalase activity is primarily the result of inorganic salt catalysis, and is not materially influenced by biological factors.

A. G. POLLARD.

Deflocculation and plasmolysis of soils. B. GANOSSIS (Compt. rend., 1928, 186, 1234—1236).—In addition to the alkali carbonates (cf. Dumont and Ganossis, B., 1928, 63), solutions of the hydroxide, chloride, or nitrate of sodium act as de-coagulants to soil and render it impermeable to water. Solutions of calcium sulphate (0.2%) or nitrate, potassium chloride or nitrate, and hydrochloric or nitric acid behave like calcium chloride (*loc. cit.*) and re-establish the permeability, the nitrate and sulphate of calcium being the most and least effective, respectively. Solutions of lime-water (saturated), potassium sulphate or hydroxide (0.1N) have no effect in the latter case, but potash applied to the soil may react as potassium carbonate. When potassium nitrate or chloride is used, there is evidence of exchange of the potassium ions with the calcium ions in the soil.

J. GRANT.

Buffering in soils. A. UHL (Forts. Landw., 1927,

2, 645; Bied. Zentr., 1928, 57, 104—106).—Jensen's method for measuring "buffer values," "buffer areas," etc. is discussed. The unsuitable development in this method lies in the determination of lime requirements, on the basis of the ratio lime added/change in p_H . Experimentally, changes in lime units are additive, whereas unit change in p_H values represents a multiplication, and the mathematical value of the fraction becomes distorted. For the purpose of determining lime requirements measurements of "hydrogen capacity," *i.e.*, the quantity of acid (or alkali) required to be added to a soil to produce a definite p_H value, are suggested. The hydrogen capacity corresponds to the maximum of the buffer formula, log acid added/acid remaining.

A. G. POLLARD.

Origin of red soils. E. BLANCK and F. GIESECKE [with A. RIESER and F. SCHEFFER] (Chem. Erde, 1927, 3, 44; Bied. Zentr., 1928, 57, 97—104).—Analyses of numerous red soils of varying types are recorded and discussed from the point of view of the correlation of chemical composition and mode of formation, age, and origin.

A. G. POLLARD.

Laboratory apparatus for the measurement of carbon dioxide evolved from soils. F. W. MARSH (Soil Sci., 1928, 25, 253—261).—In an apparatus consisting essentially of a filter flask surmounted by a cylindrical percolator and in direct communication therewith, purified air is drawn both over and through the soil, thus approximating more nearly to natural conditions.

H. J. G. HINES.

Calculation of soil respiration. H. LUNDEGÅRDH (Biochem. Z., 1928, 194, 453).—A misprint in the author's formula ("Kreislauf der Kohlensäure in der Natur," Jena, 1924, 146) for determining the production of carbon dioxide by soils is corrected, the formula becoming: $[(a - b) \times 1.858 \times V \times 60/t] / 10Y$ grams of carbon dioxide per hr. per m.² of surface, where a is the original and b the final percentage value of the carbon dioxide content of the bell-jar, t is the time in min., 1.858 the weight of 1 litre of carbon dioxide at 15° and 760 mm., V the volume of the bell-jar in c.c., and Y the surface in cm.² covered by the bell-jar.

P. W. CLUTTERBUCK.

Influence of heavy applications of dry organic matter on crop yields and on the nitrate content of the soil. A. W. BLAIR and A. L. PRINCE (Soil Sci., 1928, 25, 281—287).—Pot experiments extending over three years afford confirmatory evidence that the depressing effect of dry organic matter on crop yield is due to the locking up of available nitrogen by soil organisms.

H. J. G. HINES.

Effect of growing plants on solubility of soil nutrients. W. H. METZGER (Soil Sci., 1928, 25, 273—280).—Measurements of the bicarbonate concentration in soil in proximity to roots and in soil more remote gave higher values for the former, indicating that plants are able to exert a solvent action by excreting carbon dioxide.

H. J. G. HINES.

Manuring of the potato crop. T. J. SHAW (J. Min. Agric., 1928, 35, 36—38).—Experiments embodying the use of ammonium sulphate and potassium sulphate at four different rates, *viz.*, 0, 1, 2, and 3 cwt. per acre,

and in all possible combinations, were carried out in 1927. Each treatment was quadruplicated, and the arrangement was in four randomised blocks of sixteen plots. The experiments were set out on both dunged and undunged land. On the former, without fertilisers, the yield was 4 tons 17 cwt.; on the latter 3 tons 4 cwt. Ammonium sulphate alone, on the dunged area, gave increases of 27 cwt. and 33 cwt. for the first and second cwt., respectively, of fertiliser applied. The third cwt. of ammonium sulphate was only effective in the presence of 3 cwt. of potassium sulphate, when the mean yield reached 9 tons. On the undunged land the maximum yield was attained by the use of 2 cwt. of ammonium sulphate in conjunction with 1 cwt. of potassium sulphate, whereby a crop of 7 tons 10 cwt. was obtained.

H. J. G. HINES.

Calcium as a factor in soya bean inoculation.

R. W. SCANLAN (Soil Sci., 1928, 25, 313—325).—The general effect of calcium was to increase nodulation both in soil and in water cultures. H. J. G. HINES.

Determination of phosphoric acid. DODD.—See VII.

PATENTS.

Manufacture of fertilisers. R. C. FLUCK and A. THEIL (B.P. 289,284, 1.7.27).—Fertiliser material in faecal matter is removed by treatment with peat, lignite, etc. By plural filtration through a mass of peat, suspended matter is removed to form an organic fertiliser. Ammoniacal matter in solution may be obtained by boiling, either before or after filtration, and phosphates obtained from the liquor by precipitation. Alternatively the faecal matter may be boiled with 3—5% of peat, and stirred with an agitator. The mud after settlement may be centrifuged and prepared as a fertiliser. The clear liquor may be treated as above for preparing phosphate and ammoniacal fertilisers.

A. G. POLLARD.

Manufacture of fertilisers. E. L. PEASE (U.S.P. 1,668,464, 1.5.28. Appl., 11.3.26. U.K., 16.4.25).—See B.P. 253,291; B., 1925, 685.

XVII.—SUGARS; STARCHES; GUMS.

Desiccation (De Vecchis) process of beet sugar manufacture. C. S. GARRETT (Chem. and Ind., 1927, 46, 1014—1020).—The working of the De Vecchis process (B., 1925, 256; 1924, 991), which has been in operation in one or two Italian factories for several campaigns, is described. The beet cossettes are dried to a moisture content of about 3% at 90—100° and the heating is prolonged for 1—2 hrs. after the removal of moisture to ensure coagulation of proteins and other constituents. The dried cossettes occupy about 120—140 cub. ft. per ton, and can be stored for long periods without deterioration. The extraction of the sugar is carried out at 60—70° with the minimum amount of water in a battery of 10—12 lixiviators, and yields juices of 40—50° Brix. In open vessels, operating under gravity, extraction may occupy 4 hrs., but with closed vessels worked under pressure the period can be reduced to 2—2½ hrs., and juices of 60° Brix have been obtained. The purity of the juice is about 2.5—3.5% higher than that of the pressed juice of the original beets, which,

however, are of low quality. It is defecated at 80° with 0.25—0.3 pt. of lime per 100 pts. of fresh roots, followed by about twice as much superphosphate. Filtration yields a hard filter-press cake much smaller in amount than that from diffusion factories, and of high fertilising value. The defecation effects an increase of 2—3.2% in the purity of the juice from low-grade Italian beets. The purified juice, of 40—50° Brix, requires no multiple-effect evaporation, but is brought to 60° Brix by the re-melting of second-product sugars, and boiled to grain in the vacuum pan. The amount of molasses produced is not greater than in the diffusion process, nor are the sugar losses in exhausted cossettes and filter-press cake; also the waste waters from the exhausted cossettes are less liable to putrefaction than those from the diffusion process. It is estimated that in England the relative capital costs of plant and erection for diffusion and desiccation factories for the same annual output are about 40:26.5, and the working costs for the De Vecchis process are lower in spite of the extra fuel required for the drying process. It is suggested that the output of existing diffusion factories could be economically increased by the installation of drying plant, the present extraction plant serving, with slight modifications, for the recovery of the sugar during the period when the factories are now idle.

J. II. LANE.

Desiccation of sugar beet and the extraction of sugar.

B. J. OWEN (Rep. Min. Agric., 1927, 84 pp.).—A full report is given of an investigation into the De Vecchis process (B.P. 209,738 and 211,113; B., 1925, 256; 1924, 991) carried out at Eynsham. The conditions necessary for drying beet cossettes were examined, first in laboratory experiments and then in three types of dryers, viz., (a) a cylindrical mass dryer similar in principle to that of B.P. 235,273 (B., 1925, 648); (b) a moving-belt dryer of the type used by De Vecchis, and (c) a tray dryer with three hot-air compartments, one perforated tray with cossettes being placed over the first, another over the second, and two over the third, these positions representing successive stages of drying. Among the factors studied were the practicable thickness of the cossette layer (8—12 in.), the reduction in bulk (about 50%) and in resistance to air during drying, and the varying sensitiveness of the sugar to high temperatures at different stages of drying. It was completely established that the drying can be carried out on a large scale without inversion of sugar or caramelisation. So long as the cossettes were moist the temperature of the applied air (38—127°) had no effect on the sugar, but loss of sugar occurred from the use of high temperatures (above 113°) on cossettes having a moisture content below 20%. It was accordingly considered inadvisable to continue heating after the moisture was expelled, no benefit being found to result from the prolonged heating advocated by De Vecchis for the coagulation of proteins. The drying process could thus be completed in less than 1 hr. The tray dryer was adopted for most of the work as being easy to manipulate and control. Cossettes containing 3—5% of moisture suffered no deterioration on storage; after long exposure to the air the moisture content rose to 11—13%, but not beyond, and in large piles only the outermost layers of cossettes (to a depth

of about 6 in.) lost their original crispness. Extraction of the dried cossettes in a battery of small diffusion vessels, each provided with a calorisor for heating the juice passing from one vessel to the next, yielded juices of 45–50° Brix with purities of about 90%. These thick juices had excellent physical characteristics, being light in colour and clear, and not darkening perceptibly on exposure to air. The difficulty experienced in obtaining gravities above 50° Brix, together with microscopical evidence and osmotic experiments, led to the conclusion that the cells of the beets are not ruptured by the drying process, and that the subsequent extraction of the sugar is an osmotic phenomenon as in the case of fresh beets. A continuous diffuser in the form of a vertical cylinder with a helical conveyor to raise the cossettes against the descending juice (cf. B., 1927, 537) was also used successfully for the extraction of the sugar. Serious filtration difficulties were experienced with De Vecchis' method of purifying the juice with lime and superphosphate. Satisfactory working was attained, however, by a mechanical removal of suspended impurities in a centrifugal clarifier, either before or after liming, the precipitate produced by superphosphate being then easily filterable. The colour of the purified juice can, if necessary, be further improved by treatment with active carbons. Without the latter treatment, however, a strike of white sugar could be obtained from the juice reinforced by a proportion of second-product sugar. The amount of effluent from flumes and beet washing is, of course, the same for the desiccation process as for the diffusion process, but that from the working of the cossettes is less for the former than the latter, and would in a normal desiccation factory be distributed over a long working period. An appendix dealing with beet factory effluents describes a revolving double screen by which the coarse and finer suspended matters can be separately removed from the waste waters from flumes and washers, and some suggestions are made for the treatment of process effluent.

J. H. LANE.

Plant for the drying of cossettes in the De Vecchis process of beet sugar manufacture. G. W. RILEY (Chem. and Ind., 1927, 46, 1036–1042, 1080–1084).—The author describes the design and construction of a form of dryer intended primarily for beet cossettes, in which the material is exposed to air at a given temperature, *e.g.*, 100–105°, until most of the moisture is removed, and then to dry air at a somewhat lower temperature for a further period, *e.g.*, 1–2 hrs., to effect coagulative changes. The first stage is carried out in an upper compartment with steel wire moving belt supports for the material, whilst a lower compartment with similar belts travelling more slowly serves for the coagulation stage. The air entering is heated by direct admixture with adjustable proportions of furnace gases, and high thermal efficiency is attained by a system of recirculation in which a portion of the air most heavily moisture-laden is exhausted from the upper chamber at each circuit. The relatively dry air from the lower chamber is exhausted by fans into the furnace gases, and other fans control the admixture of furnace gases with the drying air, and the exhaust from the upper compartment. A plant drying 70 tons of beets

per day consumed in all-day runs oil fuel equivalent to 6.86–8.4 tons of coal of 13,500 B.Th.U. per 100 tons of washed roots.

J. H. LANE.

Factors influencing char filtration [of sugar solutions]. II. E. W. RICE and G. W. MURRAY, JUN. (Ind. Eng. Chem., 1928, 20, 276–277).—A portion of bone-char was washed for 3 days with distilled water, and, after drying, a solution of invert sugar was percolated upward through it, and simultaneously through a similar portion of unwashed char. On examining the filtered material, the results showed no appreciable differences for the two chars, the conclusion drawn being that the ammonia known to be present in re-activated char does not affect the selective adsorption of levulose previously observed (cf. B., 1927, 263). Adsorption tests with monocalcium phosphate were found to be vitiated owing to the presence of calcium carbonate in the char. Mixtures of sodium sulphate and calcium acetate with sugar were filtered through char, the values obtained showing an increase in the adsorption of the salt as the percentage of sugar was increased. When, therefore, the maximum of ash is to be removed by char in refining raw sugars, the liquors should be kept as dense as possible.

J. P. OGILVIE.

Influence of p_H and of lead salts on invertases, and preservation of beet pulp for analysis. E. PARISI (Annali Chim. Appl., 1928, 18, 99–103).—Vondrák's method (B., 1925, 254) of preserving beet juice and pulp by either heating them to 82° or by rendering the samples faintly alkaline by addition of lime and transporting them to the analysts at –5° to –10° is impracticable and not always effective. Change of polarisation may, however, be prevented by mixing 26 g. of the pulp or juice with 177 c.c. of 4% basic lead acetate solution. The loss in polarisation of the untreated material is due to the action of invertase, which is usually regarded as of bacterial origin, but the author finds that beet pulp from the freshly sliced roots, if placed at once in a vessel with toluene and kept tightly closed, soon undergoes a fall in polarisation. The presence of invertase in the beet, thus indicated, may play a part in the destruction of sucrose in the sliced roots.

T. H. POPE.

Influence of phosphate and colloid contents of cane juice on defecation. H. S. PAINE, J. C. KEANE, and M. A. MCCALIP (Ind. Eng. Chem., 1928, 20, 262–267).—The phosphate content of the raw juice showed an approximately linear relation to the colloid elimination. Increasing the p_H at which the juice was limed increased the colloid elimination, but also the calcium content of the defecated juice, which latter (per 100° Brix) showed a definite relation to the content of reversible colloids in the defecated juices. Excess of lime in defecation apparently has a peptising effect on the gummy substances in the juice, as there is an increase of reversible colloids proportional to the increase in calcium salts, a portion of which is of a colloidal character. By adding phosphoric acid to raw juices deficient in that constituent a decided increase in colloid elimination was exhibited, together with a distinct reduction in the calcium salts.

J. P. OGILVIE.

Factory operation of automatic electrometric p_H control of cane juice defecation. R. T. BALCH and H. S. PAINE (Ind. Eng. Chem., 1928, 20, 348—353).—Using a recording potentiometer, tungsten and calomel electrodes, continuous flow chamber, and temperature compensator for the automatic measurement and recording of p_H values, the control of the addition of the lime used for defecation has been successfully applied in a Porto Rican factory over a period of four months. If full automatic control of the liming process is not required, it is still very advantageous to adopt means of automatically recording the p_H value of the juice, using similar equipment. This could be applied to batch as well as to continuous methods of liming.

J. P. OGILVIE.

[Sugar-works' deposits.] E. O. VON LIPPMANN (Ber., 1928, 61, [B], 222—223).—Calcium citrate and calcium α -gluconate were obtained from a deposit on the walls of a sugar magazine which had been coated with lime and subsequently used for storage of inferior sucrose for a long time and at a rather high temperature. Calcium tricarballoylate was deposited in the juice preheaters during the working-up of some very unripe beet; the unusually pronounced odour of ammonia suggests that the acid was present originally as the amide or imide.

H. WREN.

Use of the Einhorn fermentation saccharimeter for the determination of sugar. T. UMBACH (Chem.-Ztg., 1928, 52, 273).—It is pointed out that as the capacity of the Einhorn saccharimeter varies from 6 to 10 c.c., whereas the graduations on the closed limb in c.c. and percentage of sugar are constant, the instrument is unsuitable for the determination of sugar in a liquid, e.g., urine.

A. R. POWELL.

Influence of the determination of the sugar content on fresh slices [of beetroot] by different methods of the total loss. A. DOLINEK (Z. Zuckerind. Czechoslov., 1928, 52, 329—333).—It is recommended that the official pipette method, employing the single normal weight of pulp and 308 c.c. of dilute basic lead acetate for the determination of sucrose in fresh slices, should be abandoned in favour of either of the following two: (1) that in which double the normal weight of pulp is treated with 354 c.c. of dilute basic lead acetate; or (2) that in which a proportionate amount of pulp is mixed with 308 c.c. of dilute basic lead acetate from an automatic pipette. Both give satisfactory results, compared with the Herles hot water digestion process, whereas if the above official method be employed in control the losses indicated may be too high by 0.13% of the sugar in the beet.

J. P. OGILVIE.

Fermentation of molasses. BIRCKNER and PAINE.—See XVIII.

PATENTS.

Manufacture of sugar. C. G. PETREE (B.P. 287,590, 18.10.26).—To reduce the viscosity of massecuite in the centrifuge and facilitate the purging of the crystals, the massecuite is heated, preferably by admixture with hot molasses, immediately before or during its introduction into the centrifuge. Apparatus for carrying out the process is also claimed.

J. H. LANE.

Oxidation of aldoses. G. B. ELLIS. From CHEM.

WORKS, FORMERLY SANDOZ (B.P. 289,280, 17.6.27).—See U.S.P. 1,648,368; B., 1928, 137.

XVIII.—FERMENTATION INDUSTRIES.

Surface of yeast as a factor in fermentation. C. RANKEN and J. R. BELL (J. Inst. Brew., 1928, 34, 265—274; cf. Ranken, B., 1927, 200).—The coating of yeast with peptone-tannin favours the production of high and coherent heads of yeast and generally retards the rate of fermentation. The amount of reproduction except in the very latest stages is diminished. The retardation is greatest during the earlier stages of fermentation, but, owing to the progressive solubility of the coating during fermentation, its effect on the time required to reach the attenuation usual in brewery practice is of small magnitude. The physical condition of the coating is influenced by traces of iron in solution which cause the yeast heads to become less coherent, with an accompanying acceleration of the rate of fermentation. With a low seeding rate, the rate of fermentation of yeast coated with calcium oxalate is retarded and the reproduction is smaller. If the amount of calcium oxalate deposit is increased, or if the rate of seeding is high, the rate of fermentation is accelerated.

C. RANKEN.

Invertase-free yeasts and their application in the selective fermentation of final cane molasses. V. BIRCKNER and H. S. PAINE (Ind. Eng. Chem., 1928, 20, 267—275).—A process in which the reducing sugars present in molasses are fermented with the production of alcohol (cf. U.S.P. 1,572,359; B., 1926, 337), thus obtaining a liquor which may be submitted to a suitable desaccharification operation, is stated to have given results that have been "fairly successful."

J. P. OGILVIE.

Drying of hops. VI. Institute of Brewing Research Scheme. Report of the sixth season's work at the experimental oast, 1926. A. H. BURGESS (J. Inst. Brew., 1928, 34, 248—264).—The time of drying is related to the air speed and depth of loading according to the expression $T = kD/a^x + M$, where T is the time of drying in min., D the depth in inches, a the air speed in ft. per min., and M is the minimum time in min. D may be substituted by the loss of water in oz. per sq. ft. of kiln floor. The colour value and aroma of the hops decrease as the temperature of drying is raised from 15° to 100°. There is a decrease in the content of α -acid when the hops are dried at 80° and above, whereas the β -fraction is unaffected by the temperature. Up to 90° the antiseptic value is unaffected, and is very slightly reduced by drying at 100°. Burning sulphur under the hops gives an inferior colour to that obtained by the use of sulphur dioxide, but the antiseptic value is higher. Sterilisation of the hops by heat and subsequent storage in the absence of air preserve and retain the antiseptic power to a greater extent than does drying.

C. RANKEN.

Conservation of the food value of barley during the process of brewing. A. A. D. COMRIE (J. Inst. Brew., 1928, 34, 284—286).—It is calculated that 100 g. of barley of calorific value 272.1 kg.-cal. will produce, after conversion into malt, 419 c.c. of beer of calorific

value 43.7 kg.-cal. per 100 c.c., i.e., a calorific value, as beer, of 183.1 kg.-cal. Accordingly, 67.3% of the human-food value of the barley is conserved in the finished beer. The sources of loss are the respiration and rootlet growth on the germinating floor, the flocculation of protein matter in the copper, and the conversion of sugar into alcohol and carbon dioxide during fermentation. C. RANKEN.

Sulphurous acid in wine manufacture. L. MOREAU and E. VINET (Ann. Falsif., 1928, 21, 130—136; cf. B., 1927, 665).—The application of sulphurous acid in the preservation of grape juice and white wines is discussed. The minimum quantity of sulphur dioxide necessary for the effective preservation of grape must is stated to be 150 mmg. of free SO₂ per litre, and of sweet white wines 40—70 mmg. according to strength.

T. M. A. TUDHOPE.

Micro-determination of phosphate in wine and other fermented liquids. G. DENIGÈS (Ann. Falsif., 1928, 21, 136—142).—A rapid colorimetric method is described. The reagent employed is a sulphuric acid solution of ammonium molybdate which has been partially reduced by the action of metallic copper. A blue colour develops on heating a solution containing phosphate ions with the reagent. The intensity of the colour is proportional to the amount of ionised phosphate present. Standards are prepared containing known amounts of phosphate. The total phosphate, including that organically combined, can be determined by first evaporating to dryness with nitric acid, and then boiling with dilute sulphuric acid. 5 c.c. of a 2% solution of the wine is sufficient for the analysis. T. M. A. TUDHOPE.

Examination of brandies. R. COHN (Pharm. Zentr., 1928, 69, 241—246, 261—265).—Methods for the determination of the alcoholic strength and ester content of spirits are described. The influence of a small amount of dissolved extract on the sp. gr. of spirits is also discussed.

T. M. A. TUDHOPE.

Adulterated spirits and the Micko distillation. C. BREBECK (Chem.-Ztg., 1928, 52, 377—379).—The Micko distillation test for detecting the origin and adulteration of spirits is discussed. The test is based on the odour and taste of the distillate prepared under definite conditions, and is the only method by which the origin of spirits can be determined. E. H. SHARPLES.

Methyl alcohol in various alcoholic drinks. W. SEIFERT (Oesterr. Chem.-Ztg., 1928, 31, 65—68, 74—77).—By the use of Denigès' method, the methyl alcohol content of a number of wines and brandies was found to be: commercial grape wines less than 0.11 to 0.25; fruit wines 0.08—0.2; wine brandies less than 0.4; fruit brandies 2.3 and 4.1; grape and fruit brandies from whole mash or residues 3.2—5.8 c.c./litre. The differences between the various classes of material examined were more marked when the proportion of methyl alcohol was calculated on the total alcohol, and the author concludes that a greater amount of methyl alcohol is present in wines from American varieties of grape and their hybrids. Experimental mashes of such grapes gave wines containing less than 0.1 to 0.58 c.c. of methyl alcohol per litre, reduced to below 0.3 by heating the mash to 70—75° before fermenting. The methyl

alcohol thus appears to be derived from the pectin, and is no longer found after the destruction of pectase by heat. In expressed must, increase of pressure slightly increases the methyl alcohol content of the wine. Some methyl alcohol may be present in the wine as esters. Literature relating to pectin and to the physiological action of methyl alcohol is reviewed, and it is suggested that brandies from American varieties of grape and from residues may contain toxic amounts. F. E. DAY.

Presence of glycuronic acid in wines from diseased or rot-infested grapes. D. CHOUGHAK (Ann. Chim. anal., 1928, [ii], 10, 97—98).—See B., 1928, 281.

PATENTS.

Purification of liquids by distillation. E. C. R. MARKS. From U.S. INDUSTRIAL ALCOHOL CO. (B.P. 287,607, 29.11.26).—High-strength alcohol is produced by rectification in the presence of benzol, the constant-boiling mixed vapour from the rectifier being separated into its constituents (which are re-used) by several stages of settling into layers and rectifications. The process is applicable to other liquids besides those mentioned.

B. M. VENABLES.

Butyl [alcohol]-acetone fermentation. D. A. LEGG, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,668,814, 8.5.28. Appl., 1.10.26).—See B.P. 278,307; B., 1928, 32.

XIX.—FOODS.

Effect of severe weathering on certain properties of wheat. W. O. WHITCOMB and A. H. JOHNSON (Cereal Chem., 1928, 5, 117—128).—Progressive weathering, as observed over a period of two years (1925—6), lowered the grade of the wheat, bleached it, and increased the proportion of damaged kernels. The test weight per bushel and the weight per kernel were decreased. The most noticeable effect was the lowering of the germination. After an initial reduction during the first two months the protein content remained practically constant. The ash of the wheats decreased during weathering, whilst that of the flours milled from them increased. The yield, pH value, and viscosity of the flour, and the quality of the gluten were unaffected. The flours from weathered wheats decreased more rapidly in viscosity during auto-digestion, but proteo-clastic activity as measured by the Sørensen titration or Van Slyke amino-nitrogen method was the same in flours from weathered wheat as in those from unweathered wheat. The colour and texture of the bread produced from weathered wheat was normal in 1925 and slightly inferior to that produced from unweathered wheat in 1926. The loaf volume showed no significant variation. Weathering reduced the fermentation time required in baking tests, and this held not only for wheat harvested at different times, but for wheat from the inside and outside of the shock.

W. J. BOYD.

Effect of delayed harvesting on quality of wheat. A. F. BRACKEN and C. H. BAILEY (Cereal Chem., 1928, 5, 128—145).—It is shown that a kernel does not change in weight when dried to a uniform moisture content after wetting, but that the decreased weight to a measured volume is due to increased volume. Microscopical examination showed that this is due to cracking of the kernel

and development of air spaces. The opacity of wheat which has been wet and the occurrence of a high percentage of broken kernels in threshing such grain are to be attributed to the presence of these air spaces and cracks. High density is shown to be associated with high protein content. Wetting produces bleaching of the grain. Titratable acidity and p_{H} showed no change as a result of delayed harvesting. Chemical and microscopical tests gave no indication of increased diastatic or proteoclastic activity. Baking tests showed no indication of deterioration as determined by loaf volume and loaf texture. It is concluded that dark hard wheat of the Turkey-red type does not deteriorate in quality through delayed harvesting apart from bleaching and increase in volume of the grain. W. J. BOYD.

Wheat and flour studies. XIV. Factors influencing the viscosity of flour-water suspensions. III. Effect of small quantities of carbon dioxide in water used for the extraction of electrolytes. A. H. JOHNSON and B. L. HERRINGTON (Cereal Chem., 1928, 5, 105—116).—The viscosities of acidulated flour-water suspensions previously extracted with water containing carbon dioxide were higher than those of similar suspensions extracted with neutral distilled water. These differences increased as the quantities of the respective waters used for extraction were increased. No greater quantities of electrolytes were extracted with water containing carbon dioxide than with neutral water. Although differences in the p_{H} value of the extracts were found, these do not explain satisfactorily the variations in viscosity. When carbon dioxide was added to the suspensions after extraction no increase in viscosity resulted. Greater quantities of protein were extracted when water containing carbon dioxide was used. It is concluded that certain proteins of wheat flour which operate to depress the viscosity of acidulated suspensions are removed more readily by water containing carbon dioxide than by neutral distilled water, and that their removal raises the viscosity of the suspension. W. J. BOYD.

Proteoclastic activity of flour. A. CAIRNS and C. H. BAILEY (Cereal Chem., 1928, 5, 79—104).—Eight chemical methods for measuring progressive proteolysis in flour suspensions were studied. Of these, Sørensen's formol titration method was found to be the most suitable. The amount of proteolysis which occurs, as measured by the amino-nitrogen formed when suspensions of high-grade flour milled from sound wheat are digested for 48 hrs. at 37°, is of the order of a few mg. per 10 g. of flour. When yeast is added the amount of proteolysis is still small, but a multiple of that which occurs without yeast. From a comparison of different flour streams from the same hard spring wheat it is deduced that the more highly refined flours milled from purified middlings will undergo less proteolysis when mixed into a dough and fermented than will the less highly refined break flours and tailings flours. Whilst flour from sprouted wheat had a much higher rate of proteolysis than flour from ungerminated wheat, the addition of 2—3% of sprouted wheat to the wheat mixture modified the proteolytic activity of the flour very slightly. Ash content and proteolytic activity tend to increase at about the same rate. No relation

between proteolytic activity and wheat variety or region of origin could be discerned. Decreased viscosity of incubated flour suspensions is associated with increased proteoclastic activity. The presence of protease in flour was demonstrated by adding safranin solution to a flour extract, collecting the precipitate, and testing its proteolytic action on casein. W. J. BOYD.

Chemistry of bread. D. W. KENT-JONES (J.S.C.I., 1928, 47, 143—149 r).—An account is given of the manufacture of bread through all its stages from the wheat to the finished product, particular attention being given to the application of chemical control. The chemistry of bread making is discussed, and the essential features of a satisfactory baking flour are stated to be sufficient diastatic activity to produce sugar for the yeast, and sufficient protein of good quality.

Standard experimental baking test [for bread]. Report of Committee of the American Association of Cereal Chemists. M. J. BLISH (Cereal Chem., 1928, 5, 158—161).—A statement of the formula, method, and equipment to be employed in carrying out this test.

W. J. BOYD.

Determination of starch in bread. P. FLEURY and G. BOYELDIEU (Ann. Falsif., 1928, 21, 124—130).—A method for the determination of starch in breads, especially those prepared for diabetic patients, is described. After hydrolysis with dilute sulphuric acid, the proteins are precipitated by means of an acid solution of mercuric sulphate, which is shown to be more effective for this purpose than lead acetate. The dextrose remaining in the solution is then determined by polarimetric or reduction methods. T. M. A. TUDHOPE.

Ripening of cheese of "Sbrinz" type. M. SOLARI (Anal. Ofic. Quim. Prov. Buenos Aires, 1927, 1, 107—140).—Analyses of "Sbrinz" cheese were made at intervals during ripening over a period of 11 months. Figures are given of the variation in content of moisture, fat, nitrogenous material (ammonia, amines, soluble and insoluble albumins), ash, sodium chloride, and acidity. A notable feature was the fall of acidity to zero after four months, followed by a rapid rise apparently connected with degradation of the fat. Ammonia, soluble albumin, and amino-compounds showed an increase parallel with the degree of ripeness. Lactose had already disappeared when the observations were started. The results are discussed. R. K. CALLOW.

Acidity in fruit juices and preserves. C. F. MUTTELET (Compt. rend., 1928, 186, 1299—1301).—The removal of pectins in fruit juices etc. before determining their organic acid content may be satisfactorily effected by first hydrolysing the pectins to pectic acid and then precipitating either the free pectic acid or its barium salt. B. W. ANDERSON.

Commercial tomato preserves. C. FERRI (Anal. Ofic. Quim. Prov. Buenos Aires, 1927, 1, 141—191).—The chemical and microscopical examination of tomatoes preserved in various forms is outlined, and a series of representative analyses is given. The essential determinations are of poisonous metals, preservatives, colouring matter, and starch. Over 6% of starchy material indicates adulteration, and sodium chloride over 1% an addition of salt. Microscopical examination

yields evidence of the efficiency of sterilisation and of the presence of other pulps, such as papaw or carrot.

R. K. CALLOW.

Changes produced in meat extracts by the bacterium *Staphylococcus aureus*. Application of the alcohol titration methods. F. W. FOREMAN and G. S. G. SMITH (Dept. Sci. Ind. Res., Food Invest., 1928. Spec. Rept. No. 31. 97 pp.).—The extract used was that of ox heart made with plain tap water and concentrated to one fourth or one tenth of its original volume. The growth of the bacteria in this medium at 37° was compared over extended periods with the variation in the proportion of amino-acids, volatile bases, volatile and non-volatile acids, etc., as determined by the alcohol titration method (cf. Foreman, A., 1928, 448). The organisms exhibited various periods of growth, each of which was characterised by a particular chemical change. In the first period the rate of growth reached a maximum on the fourth day and resulted in the production first of volatile bases, then of volatile and non-volatile acid radicals, and finally of non-volatile acid radicals alone. A second maximum occurred on the twenty-eighth day, when non-volatile acid radicals were converted into the equivalent amount of volatile acid radicals. During a third period of growth the number of organisms gradually diminished, whilst the decline in the non-volatile acid radicals continued practically to zero with production of carbon dioxide, the volatile acid radicals remaining approximately constant. During the later stages of prolonged incubation the organisms were found to destroy the volatile acid radicals at room temperature, but at 37° they attacked the amino-acids and non-volatile amines in preference.

F. R. ENNOS.

Control of reaction in cultures and enzymic digests. Comparison of the effects of certain salts on changes in p_H and changes in absolute $[H^+]$ with reference to enzyme action. Rôle of creatinine in the control of reaction in cultures. F. W. FOREMAN and G. S. G. SMITH (Dept. Sci. Ind. Res., Food Invest., 1928. Spec. Rept. No. 32, 27 pp.).—Examination of the p_H value and titratable acidity of ox-heart extracts in which *Staphylococcus aureus* has been growing for varying periods indicates that phosphates and creatinine, which are present in the ratio of 1.7:3.0, are largely responsible for the resistance to changes of p_H on addition of acid. The influence of salts on the activities of enzymes and bacteria in media should be considered from the point of view of their effect not only on the p_H values (buffer action), but also on the absolute $[H^+]$ and $[OH^-]$ (depressor effect). In the region of $[H^+]$ where phosphates have little depressor effect, the action of creatinine in facilitating the continued growth of acid-producing organisms is demonstrated.

F. R. ENNOS.

Determination of dextrose in presence of proteins [in gluten bread]. P. FLEURY and G. BOYELDIEU (Bull. Soc. Chim. biol., 1928, 10, 568—575).—See B., 1928, 383.

PATENTS.

Preparation of a pectin product. CALIFORNIA FRUIT GROWERS' EXCHANGE (B.P. 259,948, 5.10.26.

U.S., 17.10.25).—The crude pectin is rendered suitable for jellying of fruit juices either by washing with an alcoholic solution of a salt of a strong base and a weak acid, e.g., sodium bicarbonate, or by mixing with sufficient sodium bicarbonate with or without the addition of an organic acid, e.g., citric or tartaric acid, so that the finished product has a p_H value of from 3.4 up to but not including 7.0.

F. R. ENNOS.

Soluble food product having fresh kola as a base.

A. and E. CHALAS (U.S.P. 1,669,326, 8.5.28. Appl., 24.4.25. Fr., 7.5.24).—See B.P. 233,695; B., 1926, 213.

[Machines for] the manufacture of biscuits etc.

T. & T. VICARS, LTD., and E. W. CROSLAND (B.P. 289,240, 29.3.27).

Preservation of [carcases for] food. P. C.

LAVENDER and A. E. SHERMAN (B.P. 289,653, 24.5.27).

Oils containing vitamins (B.P. 289,187).—See XII.

Cans for foodstuffs (U.S.P. 1,667,212).—See XIII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Determination of potassium iodide in tincture of iodine. P. MANICKE and W. POETHKE (Pharm. Zentr., 1928, 69, 257—261).—The tincture is evaporated to dryness on the water-bath. Water is then added, and the solution is again evaporated to dryness in order to remove any hydrogen iodide which has been formed. The iodide in the residue is determined iodometrically or by Volhard's method.

T. M. A. TUDHOPE.

Esterification of ethyl alcohol in citric acid solution. C. W. CORNWELL (Pharm. J., 1928, 120, 391).—A flavouring mixture, originally containing about 25.5% of citric acid and 49.8% by vol. of alcohol, on examination after a period of 5 years, contained 38.9% of alcohol and 17.16% of ethyl citrate. In the removal of alcohol and water from a mixture containing ethyl citrate the ester is not appreciably hydrolysed, and the alcohol may be determined by direct distillation if alkali and volatile oils are absent or by direct separation with brine and distillation from a neutral solution if essential oil is present. Ethyl citrate cannot be separated from alcohol by light petroleum.

E. H. SHARPLES.

"Calcium glycerinophosphoricum solubile" and some glycerophosphates. G. KOGAN (Pharm. Zentr., 1928, 69, 49—53).—The pharmaceutical preparation "calcium glycerinophosphoricum solubile" contains in addition to calcium glycerophosphate the less soluble diglycerophosphate; the product is rendered soluble by addition of citric acid. It therefore cannot be analysed by the method of titration by acid to the acid salt, followed by back titration to the neutral salt. A preliminary titration of free citric acid (which may be detected by extraction with ether) is required; the content of diglycerophosphate is found by determination of ash (calcium meta- and pyro-phosphates). Magnesium glycerophosphate is similarly analysed; for the iron salt the iodometric method is used.

E. W. WIGNALL.

Glycerophosphates and inositolphosphates. G. LOCATELLI (Boll. Chim. Farm., 1927, 66, 737—738).—Calcium magnesium inositolphosphate, $C_6H_6O_{24}P_6Ca_3Mg_3$, is contained in the husk of cereals to the extent of about

8%, and is easily extracted by dilute acid; for pharmaceutical purposes it can advantageously replace calcium glycerophosphate.

E. W. WIGNALL.

Caucasian thuja oils. B. RUTOVSKI and K. GUSEWA (Riechstoffind., 1927, 185; Chem. Zentr., 1927, ii, 2723).—Fractionation of the oil of *Thuja occidentalis* yielded α -pinene, α -thujone, fenchone, and esters. That from the leaves of *T. Varreana* (d^{20} 0.9078, α_D -1.23° , n_D^{20} 1.4550, acid value 1.5, ester value 16.36, ester value after acetylation 30.36) yielded sabinene, α -thujone, and thujyl alcohol. That from the shoots of *T. gigantea* var. *semperaurea* (d^{20} 0.9145, α_D -1.21° , n_D^{20} 1.4552, acid value 2.34, ester value 26, ester value after acetylation 47.15) contained α -thujone, α -pinene, and thujyl alcohol.

A. A. ELDRIDGE.

Russian mint oil. B. N. RUTOVSKI and I. V. VINOGRADOVA (Trans. Sci. Chem.-Pharm. Inst., 1923, No. 4, 10—13).—The oil examined had $[\alpha]_D$ -20° to -27° , d_4^{18} 0.914—0.915; menthol (esters) 6—15%, menthol (free) 41.51%, menthone 16—18%, acid value 0.7—1.0.

CHEMICAL ABSTRACTS.

Russian fennel oil. B. N. RUTOVSKI and P. P. LEONOV (Trans. Sci. Chem.-Pharm. Inst., 1923, No. 4, 16—24).—The oil contained anethole, d -fenchone, d - α -pinene, dipentene, α -phellandrene, camphene, and anisaldehyde.

CHEMICAL ABSTRACTS.

Essential oil from *Gastrochilus panduratum*. Ridl. A. J. ULTÉE (Proc. K. Akad. Wetensch. Amsterdam, 1928, 31, 62—64).—On steam-distilling the oil from the rhizomes of the above plant and fractionating the distillate, methyl cinnamate (b.p. 255—260°) was obtained, probably to the extent of 4.32—4.46% of the oil. A fraction (b.p. 180—200°) forming about 32% of the oil gave the tests for cineole. A small fraction distilled under a reduced pressure of 35 mm. at 120—140° consisted mainly of camphor. The lower-boiling fraction from the vacuum distillation, which had been freed from cineole by shaking out with resorcinol, was repeatedly distilled from sodium in an atmosphere of carbon dioxide. An olefinic terpene was obtained as a mobile oil which had b.p. 62—64°/13 mm., d^{17} 0.8253, n_D^{17} 1.4843, α_D^{17} $+11^\circ 42'$. The b.p. at ordinary pressures, in an atmosphere of carbon dioxide to avoid resinification, was 174° at 753 mm., but it altered quickly. The liquid was not ocimene. On reduction with sodium and alcohol, steam distillation, and distillation at ordinary pressure from sodium, a fraction was obtained having b.p. 168—169°/754 mm., d^{14} 0.805, n_D^{12} 1.4553, and α_D^{12} $+12^\circ 54'$, with an odour similar to hydromyrcene. With bromine in acetic acid, the olefinic terpene gave hydrogen bromide and became coloured, but no solid compound was obtained. The lower fractions obtained by distillation of the terpene appeared to contain still another terpene.

M. S. BURR.

Apparatus for the determination of volatile oil. J. F. CLEVINGER (J. Amer. Pharm. Assoc., 1928, 17, 345—349).—An apparatus for the direct determination of volatile oil in plants and plant products consists of a round-bottomed flask containing the material mixed with water and connected with a separator and condenser. The mixture of oil and water obtained on distillation is collected in the separator, and the water is

automatically returned to the flask. Comparative determinations of the yields of oil and their physical constants obtained by the above and the U.S.P. methods show satisfactory agreement.

E. H. SHARPLES.

Absolute essence of *Salvia sclarea*. Y. VOLMAR and A. JERMSTAD (J. Pharm. Chim., 1928, [viii], 7, 390—395).—See B., 1928, 284.

Viscosity of oils. RAASCHOU.—See I.

PATENTS.

Preparation of acetylene for anæsthetising purposes. A. BOEHRINGER [C. H. BOEHRINGER SOHN] (B.P. 264,826, 17.1.27. Ger., 25.1.26. Addn. to B.P. 205,240; B., 1923, 1199 A).—Technically pure acetylene is freed from residual impurities and solvent (e.g., acetone) by passage through solid adsorbents such as active charcoal, silica gel, etc., these being pretreated with chromic acid, water, etc. if desired. Where impurities and solvent are removed simultaneously, the adsorbent should be of an acid character or be pretreated with an acid or acid substance, a small quantity of oxygen or air may be added to the acetylene, and adsorbent and acetylene should be dry.

B. FULLMAN.

Manufacture of mixed alkaloid salts. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 273,279, 14.6.27. Ger., 26.6.26).—By treating equivalent mixtures of two *Solanaceæ* alkaloids (e.g., scopolamine with hyoscyamine or with atropine) in a solvent (water, alcohol, ether) with camphoric, phthalic, meconic, malonic, tartaric, saccharic, sulphuric, or other dibasic acid, and removing the solvent in a vacuum, mixed alkaloid salts of therapeutic value are obtained.

C. HOLLINS.

Manufacture of readily-soluble organic salts of C:C-[5:5]-dialkyl- and arylalkyl-barbituric acids. ÉTABL. POULENC FRÈRES (B.P. 271,837, 14.4.27. Fr., 25.5.26).—5:5-Dialkyl- or 5-aryl-5-alkyl-barbituric acids are dissolved in water with the addition of an equivalent of a β -aminoethyl alcohol, e.g., β -amino- or β -diethylamino-ethyl alcohol, or $\beta\beta'$ -dihydroxydiethylamine. Solutions of 5—10% concentration are readily obtained and are stable below 100°.

C. HOLLINS.

Pharmaceutical product [cyclic amino-metalmercapto-compounds]. W. SCHOELLER, A. FELDT, M. GEHRKE, and E. BORGWARDT, Asss. to CHEM. FABR. AUF AKTIEN (VORM. E. SCHERING) (U.S.P. 1,667,052, 24.4.28. Appl. 1.6.26. Ger., 2.6.24).—See B.P. 234,806; B., 1925, 692.

Specific antidiabetic principle [purified insulin]. H. W. DUDLEY (U.S.P. 1,669,328, 8.5.28. Appl. 20.3.24. U.K., 27.3.23).—See B.P. 216,978; B., 1924, 654.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Viscosimeter. ALBRECHT and WOLFF.—See I.

PATENTS.

Manufacture of photographic silver halide emulsions. W. DIETERLE, O. MATTHIES, and J. REITSTÖTTER, Asss. to I. G. FARBEIND. A.-G. (U.S.P. 1,667,589,

24.4.28. Appl., 12.7.26. Ger., 7.10.25).—Substances which have been extracted from proteins by dialysis are added to silver halide emulsions during manufacture. A. R. POWELL.

Production of photo-prints and photo-copies.

I. G. FARBENIND A.-G. (B.P. 286,233, 28.2.28. Ger., 28.2.27).—Surfaces coated with salts of 1:2-naphthaquinone-4-sulphonic acid are exposed to light, *e.g.*, under a drawing on tracing cloth, and then treated with an organic compound containing one or more amino-groups. The portion of the salt undecomposed by the light is converted into a coloured anil, a positive picture being produced. B. FULLMAN.

Sodium sulphide (B.P. 289,354).—See VII.

XXII.—EXPLOSIVES; MATCHES.

Analysis of residual acid from nitroglycerin manufacture. W. YOUNG (J.S.C.I., 1928, 47,126—130 T).

—Total acidity determination does not include the whole of the nitric acid combined as nitroglycerin. For the direct determination of sulphuric acid a method is described in which nitrogen acids are distilled out in steam, the denitrated acid is oxidised with 0.1*N*-permanganate, and residual acid is titrated with *N*-caustic soda after treatment with hydrogen peroxide. A correction is applied for the alkali equivalent of the permanganate used. For total nitrogen acids the nitrometer was used and a direct method employing steam distillation is described. The latter method was also used in determining total nitrogen acids in a solution of nitrosylsulphuric acid in 97% sulphuric acid, in check work on the determination of nitrous acid in this solution by potassium dichromate. Nitrous acid in residual acid was determined by permanganate. Nitroglycerin was determined by difference between the dichromate and permanganate values and by wet combustion.

PATENT.

Ignition of explosives. W. ESCHBACH (B.P. 276,962, 30.6.27. Ger., 1.9.26).—A thermite mixture, preferably forced into caps with or without the addition of a primer, *e.g.*, nitrocellulose or thiocyanogen-perchlorate priming, ignited by a fuse or by electrical means, is used for the ignition of explosive charges. [Stat. ref.]

L. A. COLES.

XXIII.—SANITATION; WATER PURIFICATION.

Water softening with barium salts. C. J. RODMAN (Chem. Met. Eng., 1928, 35, 221—223).—The use of barium salts in softening is advantageous with waters of high sulphate content. One method is to add a determined amount of lime-water to a softener containing a large excess of (insoluble) barium carbonate. A newer process provides for the preliminary treatment with steam of a mixture of barium carbonate and lime, the material actually added to the water thus including a proportion of barium hydroxide. Barium carbonate alone will not remove more than 85% of dissolved calcium sulphate from water. The use of lime-water improves on this, but for total elimination the presence of sodium sulphate or chloride is necessary, the electrolytes precipitating barium sulphate from the

colloidal state. The second process, unlike the first, frees the water from sodium sulphate and calcium hydroxide, sodium hydroxide being formed by interaction with barium hydroxide. It has the further effect of reducing the solubility of calcium carbonate. A minimum concentration of 1 pt. of sodium chloride in 70,000 pts. of water is to be maintained for successful working. A very thin scale only is formed and priming disappears. C. IRWIN.

Chlorination of water. L. W. HAASE (Gas- u. Wasserfach, 1928, 71, 385—390).—For the purpose of this investigation freshly prepared chlorine water was used, it having been ascertained that even in the dark and at 0° it cannot be kept more than 5 days without alteration in p_H value. This value for treated water was determined electrically, the effect of chlorine on a platinum electrode being negligible for a short time. No indicators are suitable for use in presence of chlorine, but a rough alternative method is to remove the latter with its equivalent of sodium thiosulphate and titrate. The gradual increase of acidity in natural water with addition of chlorine was traced, this being due to the conversion of calcium carbonate into calcium bicarbonate. The same result is shown with water treated with calcium hydroxide of p_H 9.5—10.0. This is supersaturated with calcium carbonate and becomes turbid on being kept, but the turbidity is cleared by addition of chlorine. Water containing organic matter behaved similarly showing increased acidity, also a reduction in the oxygen absorption and an increase in the (small) permanent hardness. The organic matter acts as a catalyst. Such waters if low in lime may well be rendered corrosive. C. IRWIN.

Microdetermination of iodine in potable waters. III. Oxidation method. M. SETTIMI (Annali Chim. Appl., 1928, 18, 104—107).—Further experiments (cf. B., 1927, 958) show that microtitration of iodine with sodium thiosulphate gives satisfactory results if the dilutions of the two solutions are not greater than 0.002*N* and if the temperature does not differ greatly from the ordinary air temperature. Previous treatment of the water, or of any liquid rich in salts, with chlorine water or sodium hypochlorite leads to erroneous results. T. H. POPE.

Pollution in streams. JONES.—See II.

PATENTS.

Evaporation and distillation [of feed-water make-up]. J. S. FORBES (U.S.P. 1,666,777, 17.4.28. Appl., 26.7.21).—A stream of exhaust steam is divided, a portion being directly condensed in a main condenser; the other portion is reheated (*e.g.*, by live steam in an ejector compressor) and passes to a smaller condenser or heat-interchanger, where it is itself condensed and raises the temperature of a portion of the main warm cooling water to such an extent that the latter will partially vaporise; this additional quantity of vapour also passes to the main condenser and forms an additional quantity of feed water. B. M. VENABLES.

Settling tanks (B.P. 287,611).—See I. Preparations for respirators (B.P. 280,554).—See VII. Fertilisers (B.P. 289,284).—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JULY 6, 1928.

I.—GENERAL; PLANT; MACHINERY.

Calibration liquids for viscosimeters. D. KRÜGER (Z. angew. Chem., 1928, 41, 375).—Castor oil and glycerin are not recommended for calibrating capillary viscosimeters as they are liable to changes, the former under the influence of air and light and the latter on account of its hygroscopic nature. Solutions of sucrose in water, up to 60%, give the best results, and the absolute viscosities, in centipoise units, for such solutions at different temperatures are tabulated.

R. H. GRIFFITH.

Crushing and grinding. GROSS and ZIMMERLEY.—See VII. **Drying furnace.** ORTLEPP. **Boiler plate steels.** POMP. **Alloys in motor construction.** STEUDEL.—See X.

PATENTS.

Heating kiln. B. M. JOHNSON, Assr. to CARBORUNDUM Co. (U.S.P. 1,669,563, 15.5.28. Appl., 24.8.25).—The furnace consists of a heating compartment separated from a combustion chamber by baffles of higher thermal conductivity at the base than at the top.

H. ROYAL-DAWSON.

Grading machine. J. D. GRABILL (U.S.P. 1,661,657, 6.3.28. Appl., 10.9.27).—The openings of the endless sizing chain of a grading machine are closed by means of a flexible endless cover member which moves with the sizing chain, and is fixed thereto. F. G. CLARKE.

Screening of wet material. J. CREDO, Assr. to LOUISVILLE DRYING MACHINERY Co., INC. (U.S.P. 1,669,973, 15.5.28. Appl., 4.12.26).—Wet material is fed by gravitation on to a screen and maintained in an approximately uniform layer while passing over it. Material passing through part of the screen is collected and returned to the remaining part. L. A. COLES.

Liquid and gas contact apparatus. F. H. WAGNER, Assr. to BARTLETT HAYWARD Co. (U.S.P. 1,669,638 and 1,669,795, 15.5.28. Appl., [A] 7.2.25, [B] 17.6.25).—(A) The apparatus consists of a casing containing a number of superposed pans, each provided with a device for raising the liquid therein and spraying it in an outward direction. The gas passes upwardly around the pans through perforations in horizontal, annular baffle plates placed above the pans, at least in the lower portion of the apparatus. In (B) the perforations are provided with devices which prevent the liquid from coalescing on the under surface of the container.

F. G. CLARKE.

Catalysts for gas reactions. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H. (B.P. 263,758, 6.11.26. Ger., 29.12.25).—The exhaustion by water, carbon dioxide, sulphur dioxide, etc. of catalysts for gas reactions is

indicated by adding 2–3% of a substance which under the influence of the particular catalyst poison causes disintegration of the active mass. W. G. CAREY.

Preparation of foams for fire extinction. BRIT. DYESTUFFS CORP., LTD., and E. CHAPMAN (B.P. 289,630, 25.4.27).—Solutions of the following substances are used to assist foam formation in fire-extinguishing appliances:—sulphonates of aldehyde-naphthalene or aldehyde-phenol condensation products, alkylated naphthalenesulphonic acids, sulphonates of sulphonatable mineral oil fractions of b.p. above 200°, sulphonated alkylated mineral oil fractions, lignosulphonic acid, naphthenic acids, taurocholic acid, and sulphonated higher fatty acids. These substances are used preferably in the form of their neutral salts. C. O. HARVEY.

Hardness testing device. D. C. DAVIS (U.S.P. 1,661,718, 6.3.28. Appl., 2.2.27).—The device comprises a plunger slidably mounted in a hollow operating member which in turn moves in one direction within a casing. A compressible measuring element of known value in one end of the operating member engages one end of the plunger, at the other end of which is a test member in contact with the sample. A stop device arrests the travel of the operating member when the latter applies a predetermined compression to the measuring element. F. G. CLARKE.

Apparatus for standardising solutions. H. H. WERMINE, Assr. to BELDEN MANUF. Co. (U.S.P. 1,664,840, 3.4.28. Appl., 14.5.25. Renewed 23.2.28).—The rise and fall of a member submerged in a solution, due to changes in density of the latter, operate a valve device for supplying liquid to compensate for the changes in composition of the solution. In order to maintain the composition constant through large ranges of temperature, the submerged member has a relatively high temperature coefficient of expansion compared with that of a solid material. For this purpose a collapsible sealed capsule containing a liquid having the same coefficient of expansion as the solution may be used.

F. G. CLARKE.

Treatment of substances with heat. W. G. LAIRD, Assr. to HEAT TREATING Co. (Re-issue 16,971, 22.5.28, of U.S.P. 1,546,345, 14.7.25).—See B., 1925, 743.

Refrigerating systems or apparatus. CHICAGO PNEUMATIC TOOL Co., Assees. of R. W. DAVENPORT (B.P. 270,655, 21.3.27. U.S., 5.5.26).

Absorption refrigerating apparatus. P. M. LE ROY (B.P. 273,315, 23.6.27. Fr., 23.6.26).

Clarification of liquids (U.S.P. 1,665,167).—See XVII.

II.—FUEL; GAS; TAR; MINERAL OILS.

[Determination of] total carbon in coal. A. R. CARR and A. M. RENTE (*Ind. Eng. Chem.*, 1928, **20**, 548—549).—About 0.3—0.6 g. of coal is exploded in a bomb calorimeter and the products are expanded into a rubber balloon contained in a large bottle filled with water. The displaced water is weighed and the temperature and pressure of the gas are recorded. The carbon dioxide content of the gas is determined by measuring the percentage absorption in caustic soda solution.

J. S. CARTER.

Determination of elementary oxidisable carbon in solid fossil fuels. I. BLUM (*Bul. Soc. Romane Stiin.*, 1928, **30**, 43—49).—In determining the amount of elementary oxidisable carbon in solid fossil fuels from the quantity of carbon dioxide produced by their combustion, it is necessary to take into account the carbon dioxide originally adsorbed or absorbed in the fuel, that formed by the decomposition of inorganic carbonates, and that liberated from organic compounds under the influence of heat. The total amount of this correction may be obtained by heating a sample of the fuel in a current of nitrogen, or in a Fischer-Schrader aluminium retort. Determinations of this kind have been made with some Rumanian lignites.

R. CUTHILL.

Determination of the reactivity of coke. D. J. W. KREULEN (*Z. angew. Chem.*, 1928, **41**, 498—501).—A current of air is passed at a constant speed over a weighed amount of the coke in a porcelain tube maintained at 700°, and the amount of carbon dioxide evolved in a definite period of time is determined. The boat containing the coke, which is in the cold part of the tube while the furnace is heating up, is pushed by means of a wire into the heated portion and 2 min. are allowed for it to reach 700°. Connexion is then made to the carbon dioxide absorption apparatus (a tower containing coke moistened with 30% potassium hydroxide solution), and absorption is allowed to proceed for 5 min. The increase in weight of the tower is taken as a measure of the reactivity.

W. J. POWELL.

Characteristics of colliery surface and mine waters. N. SIMPKIN (*J.S.C.I.*, 1928, **47**, 114—115).—Attention is drawn to the need for discrimination in the use of local colliery waters for boiler or other purposes. Drainings from pit heaps and certain seams may contain considerable quantities of metal sulphates and be acid in character, e.g., 617 grains per gallon of iron, aluminium, and manganese sulphates; others may be distinctly alkaline and may be utilised to some extent to neutralise acid waters; a third class may contain sodium chloride in deleterious quantity for such purposes as washing coal for coking. Usually considerable variation in composition occurs in the different sources of water even at the same colliery.

C. A. KING.

Effect on the gas-making value of gas oil of its previous use for removal of naphthalene from coal gas. H. H. THOMAS (*Gas J.*, 1928, **182**, 538—540).—When coal gas is treated in a rotary washer with gas oil to remove naphthalene, the spent oil is usually consumed on the carburetted water-gas plant. The properties of the oil are affected both by the naphthalene

which it dissolves and by lower-boiling hydrocarbons; using 15.3 gals. of oil per million cub. ft. of gas, the sp. gr. was changed from 0.8709 to 0.8804, the flash point was lowered from 76° to atmospheric temperature, and 10% of the oil was distilled at 150° in comparison with 221° for the original sample. Gas-making tests were carried out at different temperatures in a modified form of Ross and Leather apparatus; a factor expressing the value of the oil was obtained by multiplying the volume of gas per c.c. of oil by its content of hydrocarbons, and it was evident that the value of the oil was lowered by use in the washer. Further tests were performed on the plant, using in one case new oil, and in the other a mixture of 4 pts. of new oil with 1 pt. of spent oil; by assuming a constant calorific value for blue-gas and a constant ratio of oil-gas to water-gas, the yield of therms per gallon of oil was found to be 1.0787 and 1.0218, respectively. Interpretation of these figures shows that the therm yield per gallon of spent oil is 0.7942.

R. H. GRIFFITH.

Composition of the light fractions of tar from the Kashperovka shales. G. L. STADNIKOV and A. E. VEJCMAN (*J. Russ. Phys. Chem. Soc.*, 1927, **59**, 859—866).—The light neutral oils from tar obtained by the low-temperature carbonisation of Kashperovka (Volga) shales was investigated, with a view of identifying the sulphur compounds contained in them. The shale, containing 13.4% of moisture, 46.25% of ash, and 3.8% of sulphur, was carbonised in a Fischer rotating retort, and yielded 11.1% by wt. of tar without (15% with) a current of carbon dioxide passed slowly through the mass. On straight distillation 11% by vol. came over below 140° and 36% below 200°. On removal of acid sulphur compounds with 3% formaldehyde solution in the presence of sulphonic acids, bases with 10% sulphuric acid, and phenols with 10% alkali, and distillation, the distillate between 105° and 220° was divided into six fractions, in which the sulphur content was determined and the thiophen derivatives were separated and identified by conversion into ketones by a modified Friedel-Crafts method with acetyl chloride, lead chloride being used as catalyst, owing to the prevalence of secondary reactions with aluminium chloride. The fraction 140—160° (13.1%) gave a mixture of ketones, which yielded two isomeric semicarbazones, m.p. 192—193° and 199—200°, differing markedly in crystalline form, formed probably from isomeric dimethylthiophen methyl ketones or ethylthiophen methyl ketones, and two isomeric semicarbazones, m.p. 177° and 166°, belonging to isomeric propylthiophen methyl ketones. The four *p*-nitrophenylhydrazones were also prepared, m.p. 155°, 158°, 135—136°, and 127—128°, and these confirmed the identity of the ketones.

M. ZVEGINZOV.

Continuous treatment of kerosene distillate. L. SELSKI (*Azerbaidyansk Neftyanoye Khoz.*, 1927, No. 12, 53—58).—The time of treatment is diminished by the use of an apparatus providing for atomisation of the oil, so that a large surface is exposed to the reagent and the mixing is thorough. CHEMICAL ABSTRACTS.

Determination of asphaltum in oils containing paraffin. J. MZOUREK (*Petroleum*, 1928, **24**, 403—

404).—Asphaltum in lubricating oil is determined by precipitation with benzine or with a mixture of alcohol and ether, but if paraffin is present it will be thrown out simultaneously, and has to be removed by subsequent extraction. If, however, the oil is treated with 40 times its volume of benzine and refluxed for 2–3 hrs. this difficulty is overcome, and, after cooling, the asphaltum appears as a residue. R. H. GRIFFITH.

Detection of paraffin in ceresin. D. HOLDE and K. H. SCHÜNEMANN (Z. angew. Chem., 1928, 41, 368–375).—Wax-like products from Polish and Caucasian ozokerite, consisting chiefly of isoparaffins, are refined with sulphuric acid and worked up as ceresin. Paraffin is frequently added to a ceresin, and this addition was usually detected by fractional precipitation with alcohol from chloroform solution, the fractions being examined in a refractometer; the refractive index of a ceresin is higher than that of a paraffin. A number of samples of ceresin, m.p. 60–87°, and of paraffins which may be mixed with them, m.p. 50–60°, have been examined, and it is found that the latter are less easily precipitated from solution and have much lower refractive indices except in the single case of a Rangoon paraffin. The sp. gr. of paraffins is 0.867–0.915, and of ceresins 0.912–0.943; molten paraffins are less viscous than ceresins. Treatment with chlorosulphonic or 30% fuming sulphuric acid provides still further distinction, as the extent of reaction is small with paraffins, but may involve up to 70% of a ceresin. Examination of mixtures of pure ceresin with known amounts of different paraffins showed that treatment with acids combined with precipitation from solution provided the most sensitive test for detection of added paraffin.

R. H. GRIFFITH.

Separation of crude-oil emulsions by chemical methods. T. KUCYŃSKI (Petroleum, 1928, 24, 398–403).—A water-in-oil emulsion may sometimes be broken by the addition of a third substance which is either soluble in the oil or else is added as an aqueous solution; of the first type, phenol and naphthenic acids are important. The action of the phenol is complex, as it increases the mutual solubility of the two liquids, lowers their surface tensions, and has a solvent action on asphaltums and similar substances which collect at the interface. The other group of emulsion-breaking substances consists of compounds such as soaps, and work in this connexion has led to a study of the electric charges present on the droplets of an emulsion. It was found that a natural emulsion from Boryslav-Tustanowice contained positively-charged water particles, unlike those of American oils described by other investigators. Examination of various artificial emulsions showed that when suspended in pure hydrocarbons a water droplet was always positively charged, but emulsions can exist in which the reverse conditions appear, and where different degrees of acidity or alkalinity obtain in the aqueous phase. The function of asphaltic substances in stabilising emulsions, the mutual effect of an acid and alkaline emulsion, and a number of technical methods of separation are described and the principles concerned in the various cases are discussed.

R. H. GRIFFITH.

Setting point of petroleum and residues containing paraffin. A. SACHANEN (Petroleum, 1928, 24, 654–655).—The setting point of a heavy oil depends on its previous thermal treatment, and may be altered very considerably by prolonged warming. Experiments with samples of petroleum products in small test tubes kept at any desired temperature for 1 hr. showed that a maximum setting point is developed by heating at between 40° and 60°, and an oil containing a greater percentage of paraffin will need heating at a higher temperature in order to attain its maximum. An oil or masut which contains only traces of resin or has been treated with sulphuric acid will show very slight alteration on heating; it appears, therefore, that the behaviour of an oil depends not only on the solid paraffins which are present, but also on the asphaltic substances.

R. H. GRIFFITH.

PATENTS.

Coking oven. H. H. KOPPERS (U.S.P. 1,669,168, 8.5.28. Appl., 22.12.25. Ger., 13.9.24).—The retorts are arranged in the form of a series of walls each containing a number of horizontal retorts placed one above the other, so that the heating chambers between each wall of retorts comprise vertical flues. The heating gases pass from the tops of the flues to two horizontal waste-heat collecting conduits, placed one above the other, which extend above all the flues. Below the latter are two regenerators which communicate with the bottoms of the flues.

F. G. CLARKE.

Removal of graphite incrustations from ovens, particularly coke ovens. G. CAPIAU, M. GAUQUIER, and L. LAHAUT (B.P. 289,308, 10.8.27).—The incrustation is subjected at the normal temperature of the oven to a stream of high-pressure air carrying particles of a hard granular substance, e.g., slag, which cuts away the graphite very rapidly.

A. R. POWELL.

Distillation and liquefaction of coal. F. BERGIUS (U.S.P. 1,669,439, 15.5.28. Appl., 24.11.26).—Coal is carbonised to give coke, tar, ammonia, a gaseous fraction poor in hydrogen and of high calorific value, and a gaseous fraction rich in hydrogen. The tar is mixed with a further supply of coal and is hydrogenated at an elevated temperature and under a high pressure, the gaseous fraction rich in hydrogen being utilised in this process, together with hydrogen obtained from water-gas made from the coke. The hydrogenation yields oil, ammonia, and a gas of high calorific value, which is combined with the gaseous fraction poor in hydrogen and some water-gas to give towns' gas.

A. B. MANNING.

Heat-treatment of briquettes. R. LESSING (B.P. 289,932, 31.1.27).—Briquettes are subjected to heat-treatment while embedded in a granular solid, e.g., powdered anthracite, aluminium granules, etc., whereby the danger of damaging the briquettes during the process is minimised. The temperatures employed may lie in the neighbourhood of 250°, or may approximate to those used in the carbonisation of bituminous coal. Steam or gas may be passed through the granular material to produce more uniform heating, and, if desired, a catalyst may be added to the briquette to reduce swelling of the material or binder.

A. B. MANNING.

Fuel composition. A. B. LOW (U.S.P. 1,669,341, 8.5.28. Appl., 26.1.26).—A slow-burning composition comprises finely-divided iron pyrites, a nitrate, and a small quantity of a carbohydrate. L. A. COLES.

Production of carbon black. W. K. LEWIS, Assr. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,669,618, 15.5.28. Appl., 2.1.25).—Air, heated to 500–1800°, is passed through coke so as to produce carbon monoxide at a temperature above the decomposition point of methane, and is then introduced into a thermally-insulated passage. Methane is passed into the latter in a relatively cool condition, and the products are cooled and separated. F. G. CLARKE.

Adsorption carbon. W. J. SMIT (B.P. 264,854, 20.1.27. Holl., 25.1.26).—Low-grade, unstable, adsorption carbons may be improved and stabilised by heating them with alkali hydroxides or carbonates or with calcium chloride at 200–300°, leaching out the soluble matter, washing, drying, and grinding in the usual way. [Stat. ref.] A. R. POWELL.

Activation of carbon or carbonaceous material. W. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 289,170, 24.1.27).—An apparatus for the activation of carbon comprises an inner porous fireclay tube and an outer wider iron tube. The carbon to be activated is placed either in the inner tube or in the annular space between the tubes, and steam is passed either through the annular space or through the inner tube, so that it diffuses through the fireclay walls into the activating chamber, and the water-gas produced in the process diffuses back into the steam chamber, where it is burnt to provide heat for the operation and to regenerate steam for further use. A. R. POWELL.

Production of hydrogen from gases obtained in the production of coke. R. BATTIG (B.P. 271,491, 19.5.27. Ger., 20.5.26).—The hydrogen present in coke-oven gases is separated from the other constituents by liquefying the latter. The liquefied gases are then fractionated, and a further supply of hydrogen is obtained by the thermal decomposition of the methane fraction. The decomposition is carried out by passing the methane over highly-heated coke in a generator or in the chambers of a coke oven. If the decomposition is incomplete the resulting gases may be again subjected to separation by liquefaction. A. B. MANNING.

Gas producers. WELLMAN SMITH OWEN ENGINEERING CORP., LTD., and A. V. KEMP (B.P. 289,963, 5.2.27).—The blast in a producer provided with a central tuyère surmounted by distributing hoods is regulated by means of a sleeve valve in the tuyère which is operable from the exterior of the producer. A. B. MANNING.

Gas producer plant. POWER-GAS CORP., LTD., and N. E. RAMBUSH (B.P. 290,070, 20.7.27).—The annular water-jacket surrounding the fuel bed of a gas producer is combined with the waste-heat boiler in such a way that they have a common steam drum which is connected with the water-jacket and boiler by circulating pipes. The hot gases preferably pass downward through the tubes of the boiler. In one arrangement the steam drum takes the form of an annular jacket on the upper part of the waste-heat boiler. A. B. MANNING.

Carbonising plant used in the manufacture of gas. R. W. BROADHEAD (B.P. 289,123, 17.1.27).—The gas producer is installed in a shed built on to the walls of the retort house and is connected with a dust-extracting device, such as a whirl or cyclone, which removes the dust from the gas before it enters the insulated flues leading to the regenerator. Means are provided for transferring the incandescent coke from the retort house to the producer without appreciable loss of heat. A. R. POWELL.

Manufacture of producer gas. H. L. DOHERTY (U.S.P. 1,670,102, 15.5.28. Appl., 8.9.20).—A column of fuel passes down a shaft through the sides of which air is blown, the air supply being so controlled as to maintain the highest gas-making temperatures in the upper part of the column; combustion in the lower part of the column is controlled by introducing flue gases therein in such a way that the fuel falls in temperature as it advances below the mid-portion of the column. A. B. MANNING.

Manufacture of oil-gas. C. A. PHELPS, Assr. to A. J. ANDERSON and M. G. BARMORE (U.S.P. 1,669,172, 8.5.28. Appl., 26.3.24).—A part of the oil is completely burned and the heat is utilised to volatilise and crack the remainder and also to heat up a checker-brick chamber. The volatilisation and cracking of the oil is effected out of contact with the combustion gases, but when this operation has been completed the products are mixed with the combustion gases and the mixture is passed into the heated chequer chamber where reaction takes place and a uniform combustible gas is produced. F. G. CLARKE.

Gas purification. J. BECKER, Assr. to KOPPERS CO. (U.S.P. 1,665,013, 3.4.28. Appl., 22.1.27).—Coke-oven or other fuel gas is freed from acidic impurities, such as hydrogen sulphide and hydrogen cyanide, by washing with an alkaline liquor. The latter is regenerated, after being heated, by treatment in counter-current with an upward stream of a portion of the purified gas. The fouled gas is utilised for heating the ovens or in some other part of the plant where the impurities are not objectionable. F. G. CLARKE.

Apparatus for extraction of oil from shale. R. CRAWSHAW (U.S.P. 1,666,488, 17.4.28. Appl., 5.2.27).—The apparatus comprises a heating unit which can be pushed into a mass of the shale and which is provided with pipes whereby compressed air may be forced into the shale and oil withdrawn therefrom. A. R. POWELL.

Extraction of oil from shale. W. RHOADES, Assr. to RHOADES SHALE OIL CO. (U.S.P. 1,668,820, 8.5.28. Appl., 3.12.23).—The volatile products obtained by the distillation of shale are rapidly removed from the retort by suction through a pipe which extends into the mass of shale and has adjustable openings. C. O. HARVEY.

Plant for the manufacture of liquid fuels. Soc. INTERNAT. DES PROC. PRUDHOMME HOUDRY (B.P. 267,512, 8.3.27. Fr., 9.3.26).—In processes similar to that described in B.P. 238,931 (B., 1925, 950) the whole train of catalysts etc. is suitably maintained at tem-

peratures adequate for the prevention of tar condensation in the vital parts of the apparatus.

C. O. HARVEY.

Production of liquid fuel for use in internal-combustion engines. P. G. SOMERVILLE and W. H. HOFFERT (B.P. 289,347, 26.10.26).—The rate of formation of gummy resinous substances in liquid fuels which are, immediately after refining, free from these substances, but which contain polymerisable substances, is inhibited (as shown by accelerated tests involving exposure to ultra-violet light) by the addition of small proportions of aromatic hydroxy-, nitro-, or amino-compounds; acids, aldehydes, and mercaptans appear to assist resin formation. The inhibitors employed include cresols, aniline, etc., and they must be added in the optimum proportions, due allowance being made for the phenolic and other substances already present in the fuel, the time during which the fuel is to be stored, etc. The amounts used are of the order of 0.05%; the use of more than the optimum percentage may assist resin formation.

C. O. HARVEY.

Fuel for internal-combustion engines and motors. C. A. WALTER and F. B. MUHLENBERG (U.S.P. 1,669,181, 8.5.28. Appl., 10.5.24).—The fuel consists of a petroleum product which has been agitated with ammonium chloride and to which has been added small quantities of copper and zinc sulphates.

C. O. HARVEY.

Production of a combustible charge for use in an internal-combustion engine. D. BALACHOWSKY and P. CAIRE (B.P. 265,186, 20.1.27. Fr., 27.1.26).—A hydrocarbon fuel is atomised, mixed with a quantity of air not exceeding one half of that required for combustion, and passed through a chamber heated by means of exhaust gases to 300–350° and containing a metal capable of effecting the catalytic decomposition of hydrocarbons. A further quantity of air is added before the mixture enters the cylinder of the engine.

C. O. HARVEY.

Manufacture of hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 270,705, 2.5.27. Ger., 10.5.26).—Hydrocarbons (mainly liquid) are produced by heating metallic carbonyls such as those of iron, cobalt, nickel, or molybdenum, with a proportion of hydrogen insufficient for the production of methane, and, if required, in the presence of catalysts such as finely-divided metals, alkalis, ammonia, etc. Gases containing hydrogen such as water-gas may be used, and the proportion of hydrogen may be varied according to the class of product required. Thus reduction of the proportion of hydrogen employed increases the molecular complexity of the products and *vice versa*.

C. O. HARVEY.

Manufacture of valuable hydrocarbons and derivatives thereof from coal, tars, mineral oils, etc. I. G. FARBENIND. A.-G. (B.P. 272,190, 20.5.27. Ger., 1.6.26).—The starting material is exposed, in a coherent thin layer, to the action of hydrogen at a raised temperature (*e.g.*, 450–475°), preferably under pressure (*e.g.*, 200 atm.), and, if required, in the presence of a catalyst. The material is spread in a thin layer on a screw-formed surface, corrugated band, or other device,

preferably metallic, of large superficial area, and is conveyed mechanically through the reaction chamber.

A. B. MANNING.

Conversion of coal into hydrocarbons. A. GAERTNER (B.P. 266,311, 27.1.27. Ger., 18.2.26).—Hydrocarbons are produced by injecting a mixture of powdered coal, steam, and finely-divided iron or other metal capable of producing nascent hydrogen from the steam, into a reaction chamber at 400–500° and under pressure. The iron also serves to retain the sulphur present in the mixture, and lime or other alkalis may be added to retain carbon dioxide.

C. O. HARVEY.

Apparatus for treatment [cracking] of oil. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,670,104, 15.5.28. Appl., 7.5.25).—The vapours from a battery of cracking stills are combined in a refluxing apparatus interposed between the stills and the condenser.

C. O. HARVEY.

Apparatus for treatment [cracking] of hydrocarbons. G. EGLOFF and H. P. BENNER, Assrs. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,670,105, 15.5.28. Appl., 21.8.20. Renewed 12.2.27).—Five horizontal cracking stills are arranged in a regular polygonal manner in a furnace, oil being admitted to the two lower stills and the vapours passing upwards through the next two stills and finally to a refluxing dephlegmator and a condenser *via* the fifth and topmost still.

C. O. HARVEY.

Apparatus for cracking oil. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,670,106, 15.5.28. Appl., 3.1.21. Renewed 25.3.27).—A refluxing conduit, situated in the vapour space of a horizontal still, serves for the introduction of raw oil, which, after being used as a scrubbing medium for the vapours, is passed to the bottom of the oil in the still.

C. O. HARVEY.

Conversion of heavy hydrocarbons into hydrocarbons of lower mol. wt. A. D. SMITH and J. PERL (B.P. 289,673, 12.7.27).—In a continuous process for cracking hydrocarbon oils, the deposition of carbon in the cracking plant is avoided by checking the decomposition while the highly polymerised products, which would subsequently produce a carbonaceous deposit, are still in solution, distilling off light hydrocarbons, cooling, separating from deposited matter, and then re-cracking.

C. O. HARVEY.

Heat-conversion of hydrocarbon oils. F. A. HOWARD and N. E. LOOMIS, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,670,037, 15.5.28. Appl., 28.11.21).—The oil, forced through a heated, tubular, cracking still with sufficient rapidity to prevent the deposition of carbon, passes to a lagged reaction chamber and thence to a second chamber heated by the furnace gases previously used for heating the still. A gas containing oxygen may be supplied to the second chamber.

C. O. HARVEY.

Treatment of hydrocarbons. R. T. POLLOCK, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,670,122, 15.5.28. Appl., 30.6.20. Renewed 11.4.27).—The oil is heated by passage through a number of horizontal tubes situated in a furnace fitted with a fire box and

baffle walls. The expansion chamber for the vapours is also heated by the upwardly flowing furnace gases.

C. O. HARVEY.

Treatment of oils and other similar hydrocarbons to promote cracking. F. B. DEHN. From M. B. SCHUSTER (B.P. 289,556, 31.1.27).—In the cracking of hydrocarbon oils under pressure, catalytic agents such as colloidal slaked lime or clayey substances of the bentonite type are ground and mixed with the oil to be cracked. These substances, which may require preliminary purification or treatment with dispersive agents (a hydrocarbon, water, or alcohol), are stated to be more universally useful as cracking agents than aluminium chloride, and the asphaltic residues, containing the used catalysts, are more nearly akin to natural asphalt. The catalytic mixtures may also be brought into a gel form prior to admixture with the oil.

C. O. HARVEY.

Cracking of vegetable and mineral oils. SOC. ANON. LE CARBONE (B.P. 290,060, 29.6.27. Fr., 14.5.27).—The oils are dispersed at atmospheric pressure and at relatively low temperatures (about 300–450°) through a mass of activated carbon.

C. O. HARVEY.

Cracking of petroleum oil. C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,670,103, 15.5.28. Appl., 9.3.23. Renewed 15.6.27).—The oil, after being cracked under pressure, passes to a series of expansion chambers, from which the vapours are withdrawn. The unvaporised residues from the chambers are blended in the desired proportions prior to their return to the cracking zone.

C. O. HARVEY.

Cracking of petroleum oil. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,670,108, 15.5.28. Appl., 27.10.23).—The residuum from an expansion chamber connected with a cracking still is combined with reflux condensates, and the mixture is further distilled by injection into the vapour space of the chamber.

C. O. HARVEY.

Treatment and refining of petroleum. A. M. NASTUKOFF (B.P. 289,920, 28.1.27).—Lubricating and burning oils of good colour and free from objectionable unsaturated substances liable to form asphaltic or gummy products are obtained by treating 1 vol. of crude oil with a mixture containing 4 vols. of strong sulphuric acid and 2 vols. of 40% formaldehyde, removing the more volatile products (after dilution) by means of steam, filtering, and neutralising and washing the hardened product. This product, which is in the form of a dry powder, contains considerable quantities of absorbed oil, which is removed by and recovered from cold solvents, such as benzene. The oils obtained by this process are free from decomposition products such as are produced during cracking processes.

C. O. HARVEY.

Refining of mineral oil. H. T. MAITLAND, Assr. to SUN OIL Co. (U.S.P. 1,668,920, 8.5.28. Appl., 21.12.21).—In an acid-refining process, the sulphuric acid is retained in a mass of material composed of discrete solid particles presenting a very large superficial area. The oil passes through this material and carries with it very little acid.

C. O. HARVEY.

Refining of oils. C. R. WAGNER (U.S.P. 1,669,151 and 1,669,180, 8.5.28. Appl., [A] 17.7.22, [B] 31.5.24).—(A) Petrolatum is removed from steam-refined stocks by separation after dilution with methyl ethyl ketone and chilling. (B) A mixture of butyl alcohol and naphtha containing more than 25% of the former is used in place of the methyl ethyl ketone.

C. O. HARVEY.

Refining of oils and other liquids. O. WERNER (U.S.P. 1,669,182, 8.5.28. Appl., 10.11.25. Renewed 28.3.28).—The oil is heated by passage down a column which contains a chamber heated by mercury vapour. The mercury vapour is derived from a continuous still to which condensed mercury is returned.

C. O. HARVEY.

Distillation of mineral oil. A. E. PEW, JUN., and H. THOMAS, Assrs. to SUN OIL Co. (U.S.P. 1,668,602, 8.5.28. Appl., 23.2.24).—The oil is fractionally distilled by continuous circulation through a system of stills, vaporisers, and fractionating columns.

C. O. HARVEY.

Recovery of sulphuric acid from acid sludges. J. D. RUYS, Assr. to SHELL CO OF CALIFORNIA (U.S.P. 1,669,102, 8.5.28. Appl., 18.4.27).—Acid sludge from the refining of petroleum is treated with acid oils derived from cracked petroleum oils.

H. ROYAL-DAWSON.

Treatment of emulsions. L. BURGESS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,668,941, 8.5.28. Appl., 20.3.23).—A sulphonic acid, obtained from sludge of hydrocarbons refined with sulphuric acid, is added to emulsions of hydrocarbon oils and water.

H. ROYAL-DAWSON.

Demulsification of hydrocarbons. G. EGLOFF, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,670,107, 15.5.28. Appl., 7.1.21. Renewed 4.2.27).—Caustic soda, for dehydrating purposes, is injected into the vapour space of a still in which the oil is being distilled under pressure.

C. O. HARVEY.

Deodorisation of liquid naphtha distillates and mineral oils. G. PETROFF (B.P. 289,561, 1.2.27).—Mineral oils, cracked distillates, etc. are deodorised by passing air or oxygen through the hot oil contained in a series of tanks at gradually diminishing temperatures, and containing catalysts. For instance, the oil in the first tank may contain heavy-metal salts of fatty, naphthenic, or resin acids, which activate the oxygen passing to subsequent tanks of oil which contain metal turnings such as copper or lead. The oil may require a final acid and alkali treatment.

C. O. HARVEY.

[Treatment of] mineral lubricating and transformer oils exposed at raised temperatures to air. F. HOFMANN and M. DUNKEL (B.P. 262,107, 22.11.26. Ger., 24.11.25).—Small quantities of aldehydes, and their polymerisation or condensation products with ammonia or organic amines, either alone or in admixture with one another, are added to the oils to inhibit "sludge" formation.

C. O. HARVEY.

Reclaiming used lubricating and insulating oils. A. F. MESTON, Assr. to DE LAVAL SEPARATOR Co. (U.S.P. 1,661,731, 6.3.28. Appl., 23.3.25).—The used oils are heated to 65–93°, mixed with sodium phosphate solution, and repeatedly alternately centrifuged (to separate oil and phosphate solution) and cooled to

49–60°. The oil is then mixed with 1–4% of activated earth and centrifuged.

B. FULLMAN.

Oxidation of hydrocarbons [paraffin wax]. J. R. SCANLIN, ASST. to TEXAS CO. (U.S.P. 1,668,871, 8.5.28. Appl., 20.1.26).—Heated paraffin wax is exposed to the action of an oxidising gas until the acid value of the wax is 40 or below. The wax is then free from gummy products insoluble in hydrocarbon oil.

H. ROYAL-DAWSON.

Purification of montan wax. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 289,621, 8.4.27).—The wax is treated with chromic acid in the presence of glacial acetic acid to which small quantities of sulphuric acid and/or acid salts of sulphuric or other acids are added.

C. O. HARVEY.

Protective coverings for [aircraft petrol] tanks, pipes, etc. J. POBEREJSKY (B.P. 289,659, 2.6.27).

Metal derivatives of diketones (B.P. 289,493).—See III. **Bituminous coatings** (B.P. 289,737).—See IX. **Carbons** (B.P. 289,680).—See XI.

III.—ORGANIC INTERMEDIATES.

Catalytic oxidation of aromatic hydrocarbons and their derivatives by means of air. E. B. MAXTED (J.S.C.I., 1928, 47, 101–105 r).—Many aromatic hydrocarbons and their derivatives, including those containing halogens or nitrogen, undergo oxidation when passed with air over heated catalysts containing vanadium, the most satisfactory being tin vanadate, which becomes active at an abnormally low temperature. In a series of experiments granular tin vanadate was used as a catalyst, and the ratio of hydrocarbon to air was controlled by means of an air flowmeter and by passing a measured portion of the air supply through the hydrocarbon maintained at a constant temperature. Pure benzoic acid was obtained from toluene with the catalyst at 290° (maximum yield 57.1% of the toluene vaporised); with excess of air the yield decreased, and the latter was also found to be dependent on the rate of flow over the catalyst, high rates giving much unchanged toluene whilst low rates resulted in further oxidation. Ethylbenzene gave a maximum yield of 41.3% of benzoic acid at 280°, *o*-xylene gave 59% of phthalic anhydride at 290°, whilst from benzyl alcohol 51.5% of benzoic acid was obtained at 280°. Benzaldehyde, which yielded 45% of benzoic acid at 300°, required a slightly higher catalyst temperature than toluene, which is much more difficult to oxidise, thus indicating that this temperature depends solely on the catalyst and is independent of the substance to be oxidised. Naphthalene with tin vanadate gave 84.3% of phthalic anhydride at 280°; with bismuth vanadate the maximum yield was 81.4% at 363°. Tetrahydronaphthalene yielded 52.8% of phthalic anhydride at 280°, but little or no tetrahydrophthalic acid. The yield of product in g./hr. per c.c. of catalyst space was in all cases except naphthalene about 0.02; with naphthalene it was considerably higher (0.2), because of the high relative stabilities of naphthalene and phthalic anhydride under the conditions employed. The method was unsuccessful when oxidation of *o*-cresol and toluidine was attempted, but

halogeno- or nitro-toluenes were readily converted into the corresponding benzoic acid derivatives.

W. J. POWELL.

Ethylene dichloride as solvent. K. H. BAUER and H. LAUTH (Chem. Umschau, 1928, 35, 82–86).—The chemical, physical, and physiological properties of a specimen of rectified ethylene dichloride are described. It had d_{20}^{20} 1.250, flash point (Abel-Pensky) 12–13°, and 78% of it boiled between 82.8° and 83.3°. Its rate of evaporation at 17° was comparable with that of trichloroethylene, and six times as fast as that of benzene (b.p. 76–143°). Ethylene dichloride mixes in all proportions with methyl and ethyl alcohols, acetone, ethyl acetate, benzene, toluene, light petroleum, ligroin, benzine, pyridine, carbon tetrachloride, trichloroethylene, pentachloroethane, and cyclohexanol. With carbon disulphide, or with completely dry solvents, it gave a turbid mixture. It is 0.5% soluble in water and itself dissolves 0.1% of water at 18–19°. The qualitative solubilities of a number of oils, fats, and waxes in ethylene dichloride and in its mixtures with equal volumes of ethyl and methyl alcohols are tabulated. Nitrocellulose is readily soluble in 80% of ethylene dichloride, 15% of methyl alcohol, and 5% of ethyl acetate, and even more so in a mixture with 20% each by vol. of the solvents mentioned. Cellulose acetate is equally soluble in ethylene dichloride-alcohol mixtures. Quantitative experiments show that ethylene dichloride is stable when boiled for 8 hrs. with varying amounts of water—an important consideration in connexion with the extraction of oil seeds. It is as stable to acid or alkaline permanganate as carbon tetrachloride, and much more stable than trichloroethylene, benzene, and benzene. It is stable to neutral but not to acid dichromate. A review of the literature shows that in its physiological action it is comparable with chloroform.

E. HOLMES.

Alcohol for alcoholic potash. KICZALES.—See VII. **Ethyl alcohol.** MEZZADROLI.—See XVIII.

PATENTS.

Production of methyl alcohol by catalysis. Soc. FRANÇ. DE CATALYSE GÉNÉRALISÉE, Assees. of C. HENRY (B.P. 265,948, 27.1.27. Fr., 9.2.26).—Finely-ground mixtures of 3 or 4 atoms of metallic strontium and 1 mol. of lead monoxide, or of 3 atoms of metallic zinc and 1 mol. of bismuth oxide, are used as catalysts in the production of methyl alcohol from carbon monoxide and hydrogen. Using the mixture 3Sr+PbO, the reaction is carried out at 300° and 5 atm. or 200° and 10 atm.

B. FULLMAN.

Manufacture of organic compounds [synthetic alcohols]. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 286,010, 18.8.26. Addn. to B.P. 240,955; B., 1925, 1012).—In the process and in B.P. 254,819 (B., 1926, 721) the inner parts of the apparatus with which the materials come in contact should consist of, or be coated with, metals other than iron, nickel, and cobalt, or of iron alloys stable to carbon monoxide. The catalysts and gases used must be freed from iron, nickel, and cobalt, and their volatile compounds.

B. FULLMAN.

Concentration of formaldehyde solutions. A. ZIMMERLI (U.S.P. 1,662,179, 13.3.28. Appl., 28.1.24).—Formaldehyde solutions are refluxed until equilibrium is set up between formaldehyde, its polymerides, and hydrates, and then fractionated. B. FULLMAN.

Manufacture of acetic anhydride. CONSORT. F. ELEKTROCHEM. IND., G.M.B.H. (B.P. 272,923, 15.6.27. Ger., 16.6.26).—Acetic acid vapour is heated, preferably in the presence of a catalyst, at 400–800° by passage through a vessel the heat-transfer surfaces of which are of silicon or its carbide, copper or alloys rich in it, and especially acid-resistant alloys containing chromium or nickel (chromium-iron and chromium-iron-nickel alloys). The period of heating may with advantage be shortened. B. FULLMAN.

Production of primary [aryl]amines. P. HEROLD and P. KOPPE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,662,421, 13.3.28. Appl., 20.8.27. Ger., 21.8.26).—Nitro-compounds are reduced in the presence of hydrogen or carbon monoxide, under at least 20 atm. and at 100° or over, with a solution of readily soluble sulphides insufficient in amount for complete reduction. B. FULLMAN.

Production of aromatic amines. C. W. DAVIS, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,663,476, 20.3.28. Appl., 24.4.25).—Aromatic nitro-compounds are reduced to amines by iron and water in the presence of sulphuric acid and a chloride of an alkali-forming metal. B. FULLMAN.

Purification of 3:4-dichloroaniline. I. GUBELMANN, H. J. WEILAND, and O. STALLMANN, Assrs. to NEWPORT Co. (U.S.P. 1,663,251, 20.3.28. Appl., 19.8.26).—Formation, in the presence of water, of the sulphates of a mixture of dichloroanilines permits of the separation of the practically insoluble 3:4-dichloroaniline sulphate. B. FULLMAN.

Preparation of triarylguanidines. A. E. PARMELEE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,662,397, 13.3.28. Appl., 9.4.24).—A diarylthiocarbamide is treated with an arylamine and a desulphurising agent in the presence of a chlorobenzene. B. FULLMAN.

Manufacture of phenyl-*o*-tolylguanidine. R. V. HEUSER, Assr. to A. C. BURRAGE (U.S.P. 1,669,242, 8.5.28. Appl., 27.2.23).—Aniline and *o*-toluidine in molar proportions are treated with carbon disulphide to form phenyl-*o*-tolylthiocarbamide, from which sulphur is eliminated to give the guanidine. B. FULLMAN.

Preparation of alkyl ethers of 3'-nitro-4'-hydroxy-*o*-benzoylbenzoic acid. I. GUBELMANN, H. J. WEILAND and O. STALLMANN, Assrs. to NEWPORT Co. (U.S.P. 1,665,541, 10.4.28. Appl., 28.6.26. Cf. U.S.P. 1,654,287; B., 1928, 224).—3'-Nitro-4'-halogeno-*o*-benzoylbenzoic acid is treated with an alcohol and a caustic alkali at an elevated temperature. Instead of the free acid its alkali salts may be used. F. G. CLARKE.

Production of dye intermediates [phenyl α -naphthyl ketone]. H. A. E. DRESCHER, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 289,544, 28.1.27).—*o*-Carboxyphenyl α -naphthyl ketone [*o*- α -naphthoylbenzoic acid] is decarboxylated by heating the calcium or other

alkaline-earth salt under reduced pressure at 300–330°. The phenyl α -naphthyl ketone obtained has m.p. 77°.

C. HOLLINS.

Manufacture of metal derivatives of β -diketones. BRIT. DYESTUFFS CORP., LTD., and S. COFFEY (B.P. 289,493, 28.10.26).—New metal derivatives of β -diketones containing 6 or more carbon atoms, soluble in organic solvents, and useful as antidetonants, are made by treating the diketone with a metal or its oxide, hydroxide, acetate, etc. The following derivatives are described: aluminium (m.p. 46–47°), ferric (m.p. 45°), nickelous (m.p. above 240°), cobaltous (m.p. 121–122°), cobaltic (m.p. 90°), manganic (m.p. 55–56°), chromic (m.p. 76–77°), zinc (m.p. 104), thallous, ceric, and thoracic (m.p. 63°) propionylacetones; ferric, cobaltous (m.p. 123°), and thoracic butyrylacetones; ferric, nickelous, and cobaltous γ -methylacetylacetones; aluminium (m.p. 139°), ferric, and chromic (m.p. 132°) γ -ethylacetylacetones; aluminium, ferric, and chromic γ -butylacetylacetones; nickelous acetylmesityl oxide (m.p. 140°); and manganic (m.p. 188°), chromic (m.p. 224°), and thoracic (m.p. 213°) benzoylacetones. Many of the products are intensely coloured. C. HOLLINS.

Production of derivatives of diaryl ketones. BRIT. DYESTUFFS CORP., LTD., W. H. CLIFFE, F. W. LINCH, and E. H. RODD (B.P. 289,571, 5.2.27).—The sodio-derivatives of tetra-alkyldiaminobenzophenones (B.P. 272,321; B., 1927, 598) react with compounds containing a methyl or methylene group, and the mixture of products gives on treatment with water the tetra-alkyldiaminobenzhydrol and new carbinols: $2(\text{NR}_2 \cdot \text{C}_6\text{H}_4)_2\text{CO} + 2\text{Na} + \text{CH}_2 < \rightarrow (\text{NR}_2 \cdot \text{C}_6\text{H}_4)_2\text{CH} \cdot \text{OH} + (\text{NR}_2 \cdot \text{C}_6\text{H}_4)_2\text{C}(\text{OH}) \cdot \text{CH} <$. The carbinols are readily dehydrated by boiling dilute acids to form the corresponding ethylenes. The compounds obtained from toluene, tetramethyldiaminodiphenylmethane, acenaphthene, and fluorene are described (cf. Rodd and Linch, A., 1927, 1067). C. HOLLINS.

Production of aromatic ketonic compounds. F. H. KRANZ, Assr. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,661,293, 6.3.28. Appl., 2.4.26).—4:4'-Tetramethyldiaminodiphenyl thioketone is produced by boiling under reflux, with agitation, a mixture of 10 pts. of 4:4'-tetramethyldiaminodiphenylmethane and 15 pts. of 50% sodium hydroxide solution containing 16 pts. of sulphur. B. FULLMAN.

Production of β -naphthol-1-sulphonic acid. A. E. PARMELEE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,662,396, 13.3.28. Appl., 4.3.24).— β -Naphthol is suspended in an inert liquid and treated below 16° with a solution of sulphur trioxide. B. FULLMAN.

Oxidation of ethyl alcohol. O. Y. IMRAY, From S. GOLDSCHMIDT (B.P. 290,523, 30.11.27).—See U.S.P. 1,666,447; B., 1928, 397.

Preparation of urea from carbon dioxide and synthetic ammonia. L. CASALE (M. C. SACCHI, adrix.) (U.S.P. 1,670,341, 22.5.28. Appl., 11.6.25. It., 23.12.24).—See B.P. 241,123; B., 1926, 27.

Separation of mono- and di-alkylarylamines. R. W. EVERATT and E. H. RODD, Assrs. to BRIT. DYE-

STUFFS CORP., LTD. (U.S.P. 1,670,850, 22.5.28. Appl., 9.5.27. U.K., 25.8.26).—See B.P. 273,923; B., 1927, 648.

Production of 4-nitro-2-aminophenoxy-ethanol or -propandiol [4-nitro-2-aminophenyl β -hydroxyethyl or β -dihydroxypropyl ether]. O. KNECHT, ASSR. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,669,764 and 1,670,969, 15. and 22.5.28. Appl., [A] 13.12.26, [B] 27.10.27. Ger., [A, B] 19.12.25).—See B.P. 263,191; B., 1928, 255.

Preparation of [mercaptobenz]thiazoles. L. B. SEBRELL and J. TEPPEMA, ASSRS. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,669,630, 15.5.28. Appl., 17.6.25).—See B.P. 282,947; B., 1928, 152.

Foams for fire extinction (B.P. 289,630).—See I.

IV.—DYESTUFFS.

Derivatives of sulphur dyes prepared from indo-phenols obtained from carbazole and its substitution products. E. JUSTIN-MUELLER (Sealed Notes 2126 and 2138, 6.11. and 22.12.11. Bull. Soc. Ind. Mulhouse, 1928, 94, 185—186). Report by A. RASOUMÉEFF (*Ibid.*, 186—187).—By the restricted nitration (Note 2126) of Hydron Blue R and G deep brown and olive-green dyes are obtained which yield shades very fast to washing but loose to chlorine when applied to cotton from a caustic soda-hyposulphite vat. Nitration is effected by dissolving 10 g. of Hydron R or G in 200—300 g. of sulphuric acid (d 1.842), cooling, adding a mixture of 30 c.c. of sulphuric acid (d 1.842) and 30 c.c. of nitric acid (d 1.334), and after several hours precipitating the nitro-dye by the addition of water. On prolonged nitration (Note 2138) polynitro-derivatives are obtained, Hydron Blue R yielding a dye which is more olive than that produced by restricted nitration. These nitro-dyes suffer reduction when dyed from a caustic soda-hyposulphite vat, and the resulting amino-derivatives may be diazotised and coupled on the fibre, thereby yielding shades also fast to washing but loose to chlorine. Rasoumèeff reports favourably on the processes. A. J. HALL.

Formation of lakes of alizarin. S. LIEPATOV (Textilber., 1928, 9, 496—497).—By determination with barium hydroxide of the free and combined nitroalizarin present in the barium and copper lakes obtained by treating this dye with solutions of barium and copper acetate of various concentrations as previously described (A., 1925, ii, 1059), it is shown that such lakes are not adsorption compounds, but consist of barium or copper nitroalizarates contaminated with free nitroalizarin. *E.g.*, the lakes obtained by treating nitroalizarin with 10 and 0.65% solutions of barium acetate contain 14 and 4% of barium, respectively, but both consist of barium nitroalizarate, the former being contaminated with less free nitroalizarin than the latter. It is suggested that definite chemical combination between alizarin and the mordant on cotton also occurs during dyeing. A. J. HALL.

PATENTS.

Preparation of washable aniline dyes. P. HARTMANN, ASSR. to K. SCHENZER (U.S.P. 1,662,420, 13.3.28.

Appl., 26.11.26. Renewed 25.1.28).—A permanent dye is made by evaporating to dryness mixed solutions of "aniline crystals" and boric acid. B. FULLMAN.

Manufacture of condensation products of the naphthastyril series. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 289,692, 6.9.27).—Naphthastyril is condensed in presence of phosphoryl chloride, thionyl chloride, etc. with a tertiary aromatic amine or a mono- or poly-hydric phenol. Amines give basic dyes of good light-fastness (compared with triarylmethane dyes), whilst phenols give chromable dyes; both types yield acid dyes on sulphonation. Examples are dimethyl-aniline (blue-violet, violet wool dye by sulphonation), dialkyl- α - and - β -naphthylamines (pure blue), diethyl-*m*-phenetidine (red-violet), pyrogallol (sulphonated; yellow, brilliant red-orange chromed), tetramethyl-*o*-phenylenediamine (blue-grey), tetramethyl-*m*-phenylenediamine (reddish-blue), and 1:4-dimethyl-1:2:3:4-tetrahydroquinoxaline (blue-black). C. HOLLINS.

Manufacture of triarylmethane dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 289,564, 1.2.27).—An aromatic hydroxy-compound is oxidised in alkaline solution together with a *p*-hydroxydiarylmethane, or a corresponding hydrol, or a *p*-cresol. Thus, 6: 6'-dihydroxydi-*m*-tolylmethane-5: 5'-dicarboxylic acid [methylenedi-*o*-cresotic acid] with R-acid gives a chrome reddish-blue; *p*-cresotic acid with *o*-cresotic acid (2 mols.) gives a chrome reddish-violet. The oxidant may be air in presence of copper sulphate, or lead peroxide, manganese dioxide, etc. C. HOLLINS.

Extraction of colouring matter from henna leaves. C. POLLACCHI, ASSR. to COTY SOC. ANON. SURESNES (U.S.P. 1,668,603, 8.5.28. Appl., 29.6.25. Fr. 2.7.24).—See B.P. 236,557; B., 1925, 751.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Delignification of jute fibre. J. K. CHOWDHURY and R. K. DAS (J. Indian Chem. Soc., 1928, 5, 231—243).—Phenols, in particular beech-wood creosote (Merck), remove the lignone complex from jute without affecting appreciably the cellulose. When creosote alone is used, the fibre becomes brittle, but with creosote + 10% of pyridine, the tensile strength of the product is fairly good. The effect of water at high temperatures is to reduce the fibre to a pulp, whilst excess of pyridine causes a diminution in the cellulose content. Maximum delignification with creosote + pyridine is obtained at 193—195° under 28—30 atm. for 4 hrs. The β -cellulose content of the resulting fibre is still fairly high, and it is removed partly by treatment with hot 5—6% sodium hydroxide solution. The resulting fibre retains its original colour and is of good tensile strength.

H. BURTON.

Does formaldehyde protect wool against moths? C. O. CLARK (J. Soc. Dyers and Col., 1928, 44, 144—145).—The statement of Trotman and others (B., 1928, 256) that the treatment of wool with formaldehyde would probably confer on it immunity against attack by moths is disputed. Tests at the Zoological Laboratory of the I. G. Farbenindustrie show that wool so treated has not the slightest resistance to attack by moth grubs. A. J. HALL.

Viscose. V. Quality of caustic soda, manufactured in Japan, from the point of view of the viscose industry. M. NUMA (*J. Cellulose Inst. Tokyo*, 1928, 4, 82—94).—The dissolving power of sodium hydroxide for the modified cellulose contained in pulp is diminished by the presence of sodium carbonate. Small quantities of both sodium sulphate and chloride behave catalytically in assisting dissolution, but in large quantities their action is deleterious; the presence of sulphate, even in small amount, renders the viscose film brittle. More than half the total quantity of β -cellulose in the original pulp is retained by the alkali-cellulose after removal of the excess sodium hydroxide. The most satisfactory film is given by use of a sodium hydroxide solution containing the minimum amounts of carbonate and sulphate and no insoluble impurities such as alumina, iron, silicates, etc. Sodium chloride present to the extent of 3—5% has a beneficial effect on the film. Specifications for the composition of sodium hydroxide to be used in the production of viscose are laid down.

B. P. RIDGE.

Pulping flax straw. IV. Hydrolysis and delignification with alkaline reagents. E. R. SCHAFER and C. E. PETERSON (*Pulp and Paper Mag.*, 1928, 26, 477—481; cf. B., 1927, 327; 1928, 185).—Detailed results are given for a further group of experimental cooks on flax straw using mixtures of caustic soda and sodium sulphide (4:1) at different concentrations, caustic soda alone, in admixture with, and alternating with sodium sulphite, and sodium sulphite alone, and alternating with a mixture of caustic soda and sodium sulphide. Analyses of the pulps prepared with caustic soda and sodium sulphide show that the non-lignin incrustants are removed much more rapidly than is the lignin, a total cooking time of 3 hrs. with 1 hr. at maximum temperature (155°) being sufficient to reduce the former to a minimum value. The sulphur in the cooking liquor does not react appreciably during the first 3 hrs. of the cook, though it is later consumed at a slow and fairly constant rate. The results of the other cooks indicate that (a) with caustic soda and sodium sulphite the reaction is mainly alkaline hydrolysis, though the sulphite radical does react—possibly with the products of alkaline hydrolysis; (b) with sodium sulphite alone, dissolution, particularly of the lignin, is due to the action of the bisulphite radical; and (c) the solubility of lignin in sodium sulphite solution is lowered by a previous alkaline hydrolysis, though pretreatment with sodium sulphite has no effect on the solubility of lignin in caustic soda. The chlorine requirements of the crude pulps as determined by a modification of Roe's method was in each case found to be approximately equal to their lignin content.

D. J. NORMAN.

Behaviour of lignin and chlorolignin in the preparation of wood pulp by means of chlorine. I. P. WAENTIG (*Z. angew. Chem.*, 1928, 41, 493—498).—In the chlorine method of cellulose isolation the chief economic problems to be solved are the high consumption of chemicals and the utilisation of the by-products. The chlorine attacks almost exclusively the lignin, since isolated lignin takes up about the same amount as that in natural lignocellulose, 1 pt. of lignin requiring about 1.4 pts. of chlorine, of which about 70% reappears as

hydrogen chloride. The heat liberated during the reaction is approximately 175 kg.-cal. per kg. of wood, or 120 kg.-cal. per kg. of grain straw. These results are approximate, since the amount of chlorine absorbed depends on the temperature and the time of reaction, but if these are kept constant the amounts of heat evolved, hydrogen chloride formed, and the yield of cellulose are the same whether chlorine water or gaseous chlorine (with moist wood) is used. Since the reaction is carried out at a comparatively low temperature, mechanical disintegration is desirable to facilitate penetration of the fibres by the liquor and thus ensure uniform chlorination, increase of pressure and the use of chlorine solvents being also advantageous from this point of view. In the absence of such aids the process is only suitable for straw, flax, hemp, and short-fibred woods. Though the amount of chlorine taken up increases with increasing temperature, the amount of hydrogen chloride formed increases at the same rate and the ratio remains constant. Preheating of the wood with alkalis reduces the amount of chlorine necessary, but the ratio of total chlorine to hydrogen chloride remains practically constant. If the chlorination is carried out in presence of aqueous hydrogen chloride (10—15%) instead of water, the total chlorine used is unchanged, but the amount of hydrogen chloride formed decreases, indicating that hydrolysis of chlorolignin takes place less readily under these conditions. Dry chlorine does not react with dry wood. In presence of a limited quantity of water, the temperature may rise above 100° and the concentration of hydrogen chloride in the water may exceed 20%. Both these conditions have a harmful effect on the resulting fibre, but by the use of water as a cooling agent and chlorine carrier the reaction is modified so that a strong fibre possessing good folding properties is obtained.

W. J. POWELL.

Effect on cellulose fibres of treatment in the papermaker's beater. J. W. ROWE (*Proc. Tech. Sect. Papermakers' Assoc.*, 1927, 8, 186—221).—The hydration of cellulose by beating has been studied by determining the rate of evaporation of water from unbeaten and beaten sulphite wood-pulp discs under constant drying conditions. The curves connecting rate of evaporation and percentage of water in the disc show three distinct breaks, viz., for an unbeaten pulp, at about 66% (point *a*), 20% (*b*), and 12% (*c*). Above *b* the water is considered to be free (the position of *a* depending mainly on the texture of the disc), between *b* and *c* to be held by surface forces only, and below *c* to be actually distributed amongst the cellulose molecules. The value at *c*, therefore, may possibly indicate the degree of hydration of cellulose, and on this basis experiments show that whilst hydration increases with beating, the beaten pulp "dehydrates" when left in contact with water for a long period, though even when "dehydration" is almost complete, as indicated by the position of *c*, the pulp would still be considered "wet" from the papermaker's point of view. In explanation of this "dehydration" effect and of the action of the beater in general, it is suggested that beating effects a partial conversion of the cellulose from a crystalline into an amorphous state, and that, on keeping, the amorphous (hydrated) variety tends to revert to the

crystalline form. Some support is lent to this theory by the observation that the viscosity of sulphite pulps in cuprammonium hydroxide solution is not lowered by any degree of beating that would be used in the manufacture of paper, which precludes the theory that the hydration of cellulose by beating is due to an increased dispersion of the cellulose aggregate. D. J. NORMAN.

Recent work on the oxidation of cellulose. J. L. PARSONS (Ind. Eng. Chem., 1928, 20, 491—493).—A review of recent work on the degradation of cellulose lends support to the view that the term "oxycellulose" cannot be applied to any individual chemical compound. "Oxidised cellulose" is to be preferred when referring to the products of the complex reactions involved in the oxidation of cellulose. D. J. NORMAN.

Cellulose resources. I. Annual wood crop. G. M. ROMMEL (Ind. Eng. Chem., 1928, 20, 494—496).—The estimated yield in cub. ft. and tons per acre per year at 30 and 60 years of age which may be expected from some of the common trees found in American forests is tabulated. The southern pines grow to pulpwood size in 15 years as against 30—50 years for northern pines, and, if an economical method of removing resin could be found, the southern pines would be available for newsprint. The possibilities of timber waste and sawmill wood-waste for pulpmaking are also outlined. D. J. NORMAN.

Bamboo. I. Composition of the bamboo "Mōsō-Chiku." Y. UEDA, K. KASAMA, and K. KIMURA (J. Cellulose Inst. Tokyo, 1928, 4, 95—98).—The Japanese bamboo "Mōsō-Chiku" contains (approx.) water 8—9%, cellulose 42%, lignin 24.5%, pentosan 23%, alcohol and benzene extract 3%, the cellulose obtained from the dried product by the chlorination method having the following characteristics: furfuraldehyde yield 4%, copper value 0.7, α -, β -, and γ -cellulose content, respectively, 79%, 18%, and 3%. The yields by acetolysis and sulpholysis are 27 and 80%, respectively, whilst hydrolysis by 7% sulphuric acid gives xylose crystals amounting to 3.7% of the weight of dry cellulose taken. B. P. RIDGE.

Bamboo [for papermaking]. W. RAITT (Proc. Tech. Sect. Papermakers' Assoc., 1927, 8, 89—98).—The claims of bamboo as a practically inexhaustible source of paper pulp are advanced. The earlier difficulties of preparing bleached bamboo pulp have been overcome and the waste due to nodes has been eliminated by crushing. By adopting fractional digestion in conjunction with the sulphate-soda process it is possible, using 16% of soda for 5 hrs. at 24 lb./in.², to obtain a 45% yield of pulp requiring only 8% of bleaching powder (yield of bleached pulp 42%). It is estimated that unbleached bamboo pulp could be delivered (c.i.f. British ports) at £11 10s. per ton. D. J. NORMAN.

Shortening of the time of cooking in the Mitscherlich process [of paper manufacture]. L. FRIEDLÄNDER (Papier-Fabr., 1928, 26, 335—337).—In place of the usual heating coils in the cooker an internal, cylindrical, copper heating chamber is recommended. The chamber is fitted with heating tubes so arranged that the inside walls of the chamber and the outsides of the tubes are heated by steam, whereas the liquor is heated

inside the tubes and by the outside walls of the cylinder. In this way a considerably increased heating surface is obtained with circulation of the liquor through the tubes, and the time of cooking is halved. B. P. RIDGE.

Ethylene dichloride as a solvent. BAUER and LAUTH.—See III. **Bleaching of cellulose.** RYS.—See VI. **Degumming power of soaps.** TSUNOKAE.—See XII.

PATENTS.

Manufacture of a substance to be used in laundries. H. II. BOCKWEG (B.P. 281,998, 2.8.27. Holl., 8.12.26).—By the use of a mixture containing 75—150 pts. by wt. of powdered ultramarine, 3—15 pts. of powdered dragonblood, and 1000—2500 pts. of sodium bicarbonate, it is claimed that laundry operations may be reduced in number, and that an economy of 50—60% of chlorine and 15—35% of soap may be effected. E. HOLMES.

Treatment of fibres, fabrics, etc. made of or containing cellulose derivatives. BRIT. CELANESE, LTD. (B.P. 260,290, 22.10.26. U.S., 24.10.25).—The material is treated with a solution of tannic acid of sufficiently high concentration, e.g., about 30%, to exert a swelling effect on the cellulose derivative (acetate). The solution is preferably maintained at 50—70°, and the duration of the treatment may be $\frac{1}{2}$ —2 hrs. The tannic acid thus introduced is preferably, but not necessarily, fixed by a further treatment with a solution containing tartar emetic or stannic chloride. The weighted silk shows increased resistance to heat and to slipping and laddering. D. J. NORMAN.

Treatment of fibrous materials for pulping purposes. T. L. DUNBAR, Assr. to CHEMIPULP PROCESS, INC. (U.S.P. 1,669,234, 8.5.28. Appl., 22.4.27).—Steam is introduced into the digester during the cook, and is allowed to condense and mix with the digestion liquor. Continuous circulation is maintained, and when the mixture reaches a predetermined volume the excess of liquor is transferred to an accumulator. D. J. NORMAN.

Manufacture of cellulose esters. Cellulose acetate. H. L. BARTÉLEMY, Assr. to RUTH ALDO Co., INC. (U.S.P. 1,668,482—5, 1.5.28. Appl., [A—C] 27.1.28. [D] 30.1.28. Fr., [A] 28.12.27, [B, C] 29.12.27).—(A) Dilute acetic acid containing hydrochloric and hydrofluoric acids is used for the partial hydrolysis of the primary acetate resulting from the acetylation of cellulose in the presence of a catalyst. (B) Esterification of cellulosic fibres is facilitated by pretreating them with a mixture of gaseous acetic acid and a halogen. (C) The esterification of cellulose or its conversion products is effected by a series of at least four successive partial acetylations, each of which is completed before the succeeding one is commenced. (D) Cellulosic material is treated first with an alkaline solution containing peroxides and a soap and then as in (B). Acetylation and hydrolysis are conducted as described in (C) and (A) respectively. D. J. NORMAN.

Esterification of hydrated cellulose with lower fatty acids. Reacetylation of cellulose acetate. H. T. CLARKE and C. J. MALM, Assrs. to EASTMAN

KODAK Co. (U.S.P. 1,668,945—6, 8.5.28. Appl., 12.1.27).—(A) The hydrated cellulose is treated at 100—170° and in the absence of a catalyst with a fatty acid having more than one and less than eight carbon atoms until 4 mols. of the latter combine with 1 mol. of the former, assuming the cellulose molecule to contain 24 carbon atoms. (B) Cellulose acetate, the solubility of which has been reduced by the loss of acetyl groups, is treated with acetic acid, as sole acetylating agent, at a temperature below the decomposition point of the product until the original solubility is restored.

F. G. CLARKE.

Reducing the viscosity of nitrocellulose materials. C. U. PRACHEL, ASSR. to EASTMAN KODAK Co. (U.S.P. 1,661,736, 6.3.28. Appl., 27.2.25).—Solid colloidal nitrocellulose materials are treated at atmospheric pressure with an aqueous mixed mineral acid solution, containing nitric acid, of insufficient strength to increase the nitration.

B. FULLMAN.

Manufacture of artificial silk threads. H. WADE. From N. V. BOUWONDERNEMING KETABANG IV (B.P. 289,976, 9.2.27).—The length of travel of viscose filaments in the coagulating bath is increased to such an extent, *e.g.*, from 8 in. up to 24 in. for a sulphuric acid-sodium sulphate bath, that coagulation and all further normal alterations that the filaments may undergo are practically completed within the bath itself. The filaments are then twisted and wound on a frame.

D. J. NORMAN.

Recovery of soda ash [from digestion of cellulose]. J. HOLMES, H. A. KINGCOME, and J. L. JARDINE (B.P. 288,699, 11.1.27).—The spent liquor from the digester is discharged into a closed receiver and is fed therefrom into a waste-heat boiler where it undergoes a preliminary concentration. After further concentration in an evaporator, the liquor is sprayed into an incinerator and burnt, the heat from this operation being used to heat the waste-heat boiler, which in turn supplies the low-pressure steam requirements of the digesters and soda recovery plant. To facilitate the operation of the waste-heat boiler, vigorous circulation should be maintained therein by mechanical means (*cf.* Holmes, B., 1928, 120).

D. J. NORMAN.

Cooking of wood to form chemical paper stock. W. H. HOWELL, JUN. (U.S.P. 1,670,156, 15.5.28. Appl., 5.3.25).—The spent liquor withdrawn from the digester after completion of the cook is immediately subjected to a vacuum to concentrate it, and is at the same time indirectly heated by steam from the digester.

D. J. NORMAN.

Safety paper. W. R. ORNDORFF, ASSR. to TODD Co., INC. (U.S.P. 1,662,509, 13.3.28. Appl., 8.1.25).—The paper contains pyrogallol 1 : 3-dimethyl ether.

B. FULLMAN.

Manufacture of artificial silk. C. M. WHITTAKER, ASSR. to COURTAULDS, LTD. (U.S.P. 1,670,919, 22.5.28. Appl., 1.6.26. U.K., 27.7.25).—See B.P. 254,531; B., 1926, 782.

Fire-proofed [cellulose ester] products E. C. R. MARKS. From DU PONT VISCOLOID Co. (B.P. 289,936, 1.2.27).—See U.S.P. 1,633,067; B., 1927, 775.

Separation of bark or bast from plant stalks. E. L. A. HEIMLICH (B.P. 265,618, 5.2.27).

Treatment of fats and oils (B.P. 281,232).—See XII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Bleaching of sulphite-cellulose. L. RYS (Papier-Fabr., 1928, 26, 256—260, 269—275, 288—289).—The advantages of bleaching in higher-stuff density (increase of reaction velocity, cold bleaching, greater space efficiency, lower power consumption, etc.) are discussed, and it is shown that by a combined chlorine water-hypochlorite bleach and alkali extraction an unusual degree of purity of colour, good chemical properties of the material, and about 30% economy of chlorine are obtained. The duration of the alkali extraction depends on the temperature and alkalinity of the liquid, milk of lime giving as satisfactory results as sodium hydroxide. In the pre-bleaching about 75% of the required total chlorine is used as chlorine water, the remaining 25%, as hypochlorite, being used in the after-bleaching, which may be catalytically accelerated by the use of metallic chlorides (*e.g.*, of copper, cobalt, or nickel). The economy of chlorine is ascribed partly to the preliminary alkali extraction and partly to the use of chlorine water. By this method the α -cellulose content is never lower, and the copper number little, if any, higher than corresponding values given by other processes, whilst a better colour is obtained. The relationship between the different degrees of fluorescence in ultra-violet light and the purity of the bleached material is established, comparisons being made both visually and photographically. Pure cellulose has a bluish-violet fluorescence, whilst the less pure is the material the more its colour approaches greenish-grey to brown. Theoretical and practical grounds for bleaching with hypochlorite in low and high stuff density, and for the chlorine water bleach and alkali extraction of the chlorinated incrusting substances are discussed.

B. P. RIDGE.

Apparatus for the analysis of solutions, especially bleach liquors containing chlorine. K. HINTZMANN (Chem. Fabr., 1928, 266—267).—To determine the available chlorine in a bleach liquor an apparatus comprising a graduated tube with a lower reaction bulb and an upper stoppered bulb holding 75 c.c. of liquid is used. The reaction bulb is filled with 75 c.c. of a standard solution of indigo-carmin (1.46 g. of the 44/45% powder and 2 c.c. of sulphuric acid/litre), the stopper placed in the upper bulb, the apparatus inverted, and the burette read. The whole is turned back to the normal position and the solution to be analysed is added slowly until the colour of the mixture changes from blue to light yellow-green. The stopper is replaced, the apparatus inverted, and the burette again read to obtain the amount of bleach liquor added.

A. R. POWELL.

Discharging indigo-dyed fabric by means of nitric acid. (A) Action of certain catalysts. (B) Discharges with Flavanthrene and Thioindigo Red B. (C) Discharges with nitroso- β -naphthol. N. VOSNESSENSKI (Sealed Notes 1924—5, 2.9.09, and

1940, 18.10.09. Bull. Soc. Ind. Mulhouse, 1928, **94**, 187—189, 189—190, 190—191). Report by L. PAULUS, (*Ibid.*, 191—192).—(A) In the presence of small quantities of catalysts such as potassium sulphite, sodium bisulphite or hyposulphite, powdered aluminium, zinc, or copper, and particularly sodium nitrite, the discharge of indigo-dyed fabric may be effected by means of nitric acid, using a lower concentration of acid and temperature of treatment than would be required otherwise. For example, a mixture containing 100 g. of sodium nitrate and 20 g. of sodium nitrite produces discharges equal to those obtained with 200 g. of sodium nitrate, and sulphuric acid, (d 1.261) may be used instead of acid of d 1.372. (B) Coloured discharges on indigo may be obtained with dyes of the flavanthrene and thioindigo series, very fast to light and washing, provided that the discharge is effected by means of nitric acid instead of the more usual chromate method. (C) Clear olive discharges on indigo grounds, not obtainable by the use of albumin colours and the usual chromate method of discharge, may be produced by the nitrate discharge using nitroso- β -naphthol and iron or chromium nitrate, and a basic dye if desired. Paulus confirms the originality and usefulness of the above processes. A. J. HALL.

Effect of light on indigo-dyed [cotton] fabric. R. HALLER, J. HACKL, and M. FRANKFURT (Textilber., 1928, **9**, 415).—Claim is made for the independent discovery (cf. Hibbert, B., 1927, 840) that isatin is formed during the fading of indigo-dyed cotton by exposure to sunlight. During exposure under glass for 4 months indigo-dyed fabric changed to an olive colour, and the faded fabric yielded yellow extracts with hot water or dilute alkalis; the aqueous extract gave a blue colour when treated with pyrrole and sulphuric acid, and formed crystals (m.p. not determined) identical with those of isatin. The faded portions of the fabric also contained oxycellulose which was sufficient to reduce indigo in the presence of caustic soda. It appears that the fading of indigo is a process of oxidation, and is thus in marked contrast to the fading of cotton dyed with Indanthrene Yellow. Under similar conditions of exposure undyed cotton shows no appreciable deterioration, so it is concluded that the indigo assists the oxidation of cotton during exposure to light. A. J. HALL.

Theory of leather dyeing. H. SALT (J. Soc. Dyers and Col., 1928, **44**, 134—135).—Acid and direct dyes, but not basic dyes, are readily absorbed by chrome leather, but if the chrome leather is tanned with vegetable tannages before dyeing its affinity for acid and direct dyes is reduced and its affinity for basic dyes increased proportionally to the amount of tannin fixed by the hide. Vegetable-tanned leather has a considerable affinity for basic dyes, this affinity being reduced by treating the leather with basic chromium salts before dyeing. These facts support the theory that the acid groupings of hide are "occupied" (neutralised) by mineral tanning agents, and the basic groupings by vegetable tanning agents. The usual preliminary treatments of hides before tanning increase the reactivity of the acid and basic groupings and so increases the affinity of the hides for dyes. It is suggested that this affinity of a vegetable leather for acid dyes is

due to partial replacement of the vegetable tannic acid combined with the hide by the acid dye, and since the leather-colour acid compound is insoluble, gradual exhaustion of the dye liquor occurs. If the vegetable tannic acid is fixed in the hide by drying or by treatment with tartar emetic before dyeing, the hide has no affinity for acid dyes. The degree and rate of replacement of vegetable tannic acid by an acid dye is dependent on the acidity of the dye liquor, considerable and rapid replacement occurring in a liquor of p_H 2, so that the leather is superficially and unevenly dyed, whereas in a dye liquor of p_H 6 the affinity of the leather for acid dyes is small, so that the resulting dyeings are even and well penetrated. It is usual to obtain less even shades by dyeing chrome leather with acid or direct dyes than by dyeing vegetable leather with basic dyes, but more even and better penetrated dyeings may be obtained by the former method provided that the acidity of the dye liquor is maintained at about p_H 6 by the suitable use of sodium or ammonium acetate and sulphuric acid in the dye liquor. Indigosol and Soledon dyes (water-soluble esters of leuco-vat dyes) may be dyed on leather by the methods used for acid dyes.

A. J. HALL.

Wetting-out and emulsifying agents. A. NOLL (Papier-Fabr., 1928, **26**, 318—326).—The properties of a number of commercial preparations for use as wetting-out and emulsifying agents are described. Comparisons are made between their percentages of ash, its sulphate content, the percentage of the dry substance both extracted by and precipitated by alcohol from aqueous solution, and the colours of the fluorescence of their aqueous solutions. The stability of the substances is examined by treating one part by wt. of the compound and of a cyclic alcohol or ketone (*e.g.*, cyclohexanol or cyclohexanone), with one or more parts of water and observing the conditions under which a non-homogeneous mixture is obtained; the results of the examination are tabulated.

B. P. RIDGE.

Coloured reserves under sulphur dyes [by printing]. H. FLEICHER (Sealed Note 1941, 23.10.09. Bull. Soc. Ind. Mulhouse, 1928, **94**, 195—197). Report by V. SCHWARTZ (*Ibid.*, 197—198).—A process for obtaining coloured reserves under sulphur dyes consists of printing on cotton fabric previously prepared with a 3.5% solution of potassium ferricyanide, a reserve paste containing a solution of a basic dye in phenol, a solution of zinc tungstate dissolved in any suitable organic acid, zinc chloride, and a thickening consisting of gum Senegal and china clay, then drying, steaming in a Mather-Platt for 4 min. at 102—108° (the most satisfactory conditions of steaming are those commonly used for discharging azoic colours with Rongalite), and afterwards padding with a sulphur dye liquor, oxidising by means of an "air run," and immediately washing in cold water, souring with sulphuric acid (d 1.036) at 50°, washing with cold water, and soaping. Schwartz reports that the resulting coloured reserves are much faster to washing than those obtained previously by methods in which the basic dye is fixed with zinc ferro- or ferri-cyanide alone, this increased fastness being due to the additional presence of zinc

tungstate which is capable of forming fast lakes with basic dyes. The process suffers from the disadvantage that the reserved portions of the cotton fabric are tendered by the tartaric acid used in the reserve paste, but this may be avoided by adding sodium tungstate to the "prepare" liquor and omitting zinc tungstate from the reserve paste, the use of tartaric acid thus being rendered unnecessary.

A. J. HALL.

Production of [coloured] photographic prints on cellulosic materials [cotton fabrics]. M. MICHELS (Bull. Soc. Ind. Mulhouse, 1928, 94, 124—135). Report by P. BRAUN (*Ibid.*, 135—138).—A continuous and economic process for producing coloured impressions from photographic negatives on cotton and especially viscose silk fabrics consists of padding the fabric with a sensitising solution (100 g. of green ammonium ferric citrate, 50 g. of silver nitrate, 30 g. of an organic acid, *e.g.*, tartaric, citric, or lactic acid, and 1 litre of water), drying, exposing the fabric under a photographic negative to light, washing with water, treating with a 1—2% solution of sodium chloride to convert unremoved silver nitrate into silver chloride, again washing with water, and treating with a solution containing lead ferricyanide and acetic acid, whereby silver present in the image on the fabric is converted into a mixture of the ferrocyanides of lead and silver which, after washing and treatment with a solution of cupric chloride, are converted into cupric ferrocyanide and the chlorides of lead and silver. After further treatment to remove silver chloride the fabric is immersed first in a solution containing sodium sulphite, sodium carbonate, and 1% of quinone (a catalyst), whereby the cupric ferrocyanide of the image (brown) is reduced to cuprous ferrocyanide (nearly colourless), and then for 10—15 min. in a solution containing acetic acid and a basic dye at 80—90°; washing with warm water, soaping, again washing, and then drying completes the process. About 98% of the silver used is recovered. Braun reports favourably on the process since the resulting coloured images have excellent fastness to light and washing, the shades being equal in brightness to those obtainable by means of basic dyes on a tannin mordant. Using an ordinary photographic negative an exposure of $\frac{1}{2}$ —1 hr. in full sunlight or $\frac{3}{4}$ —1½ hrs. under a mercury vapour lamp was necessary to obtain satisfactory prints on cotton fabric.

A. J. HALL.

Colouring of rubber. SANDERSON.—See XIV.

PATENTS.

Dyeing of pelts, hairs, feathers, and the like. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 289,730, 30.11.27).—The pelts etc. are treated in successive baths first with a *m*-aminophenylnaphthylamine salt and then with nitrous acid. Examples are *m*-aminophenyl- β -naphthylamine (yellow to pink), 2'-amino-*p*-tolyl- β -naphthylamine (yellow to pink), 7-(2-amino-*p*-tolyl)amino- β -naphthol (reddish-brown). The last-mentioned compound is obtained from 2:7-dihydroxynaphthalene and *m*-tolylenediamine in presence of bisulphite.

C. HOLLINS.

Dyeing of animal fibres, textile fabrics, or substances of a protein nature. S. W. WILKINSON (B.P. 289,578, 12.2.27. Cf. B.P. 242,027; B., 1926, 11).—

The affinity of wool, silk, etc. for dyes is increased by treatment with an oxidisable acid, *e.g.*, nitrous or hypochlorous acid, followed by ozone or ozonised air.

C. HOLLINS.

Dye-printing of fabrics with a free non-repeat design. R. S. BRACEWELL, Assr. to MARVELLUM Co. (U.S.P. 1,668,934, 8.5.28. Appl., 18.5.25).—A fluid mixture of oil and a finely-divided insoluble dye capable of giving a soluble leuco-compound is distributed on the surface of water or other non-solvent liquid to form a floating film of mutable design. The fabric is brought into contact with the colour film, and the colour is then reduced on the fibre to its leuco-compound, which penetrates the material and is finally re-oxidised.

C. HOLLINS.

Apparatus for the wet-treatment of textile fibres and yarns in hanks. O. SINDL (B.P. 284,984, 11.5.27. Ger., 7.2.27).

Dyeing and like machines. C. S. BEDFORD (B.P. 290,138, 31.12.27).

Bleaching of fur skins (U.S.P. 1,668,875).—See XV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Contact process for sulphuric acid. W. H. DE BLOIS (Trans. Canad. Inst. Min. and Met., 1927, 30, 929—949).—A review of the development and principles of the contact process for the manufacture of sulphuric acid with a brief description of the application of the process to the gases produced in the bessemerising of nickel-copper matte at Sudbury. The gas from the converters contains about 14% SO₂, but is diluted with air before passing through the purification plant. The catalyst used is platinised asbestos, and, by the use of heat exchangers, the heat of the issuing gases from the catalytic chamber is sufficient to preheat the incoming gases nearly to the requisite temperature.

A. R. POWELL.

Contact process for the manufacture of sulphuric acid from zinc blende roaster gases. S. ROBSON (Trans. Canad. Inst. Min. and Met., 1927, 30, 950—981).—The roaster gases obtained from zinc ores containing more than 2% Pb carry a large quantity of lead sulphate fume as well as arsenic and other impurities. These are removed in modern plants by passing the gases through a series of baffled dust-settling chambers, thence through Glover towers and cooling pipes to an electrostatic separator, from which they are passed through coke filters and washing and drying towers to the contact plant. For efficient working of the electrostatic separators the temperature of the gases should not exceed 500° and the velocity must not be greater than 1 ft./sec. Numerous modern types of washing, drying, and purifying towers are illustrated diagrammatically, and brief notes of their method of working and efficiencies with various impurities are given. The conversion is effected in two vessels, each of which is provided with a heat-interchanger. The first converter operates at a higher temperature (*e.g.*, 530°) than the second, and converts the greater part of the sulphur dioxide into trioxide at a rapid rate; the second converter, serving to effect complete conversion,

is required only in districts in which sulphur dioxide discharged into the atmosphere will affect surrounding industries. Magnesium sulphate is the most suitable contact mass for high temperatures, and asbestos for the lower temperatures.

A. R. POWELL.

[Mechanical] developments in the potash industry. R. EHRLHARDT (Chem. Fabr., 1928, 277—278).—The much larger outputs which are necessary to render potash manufacture profitable to-day have involved alterations in the methods of handling. Wagons of crude salt are now moved and tipped mechanically. The salt is broken in mills in which it falls upon beaters striking upwards. These produce grains of about 4 mm. diam. with little fines, and are built with throughputs of up to 250 tons per hr. With high outputs it is better to reduce to a certain size in beater mills and to finish the crushing with rollers. To economise power, the feed to these secondary mills is passed through a vibrating sieve, one design of which is described and illustrated.

C. IRWIN.

Purification of alcohol for the preparation of alcoholic potassium hydroxide. S. KICZALES (Ind. Eng. Chem., 1928, 20, 493).—2.5—3 g. of lead acetate dissolved in 5 c.c. of distilled water are thoroughly mixed with 1 litre of alcohol and to this mixture is added, without stirring, a solution of 5 g. of potassium hydroxide in 25 c.c. of warm alcohol. After 1 hr. the whole is well shaken, allowed to settle, and the decanted liquor filtered and distilled. This method is more efficient than the silver oxide method owing to the fact that the lead oxide-aldehyde additive product is less soluble than the corresponding silver oxide product.

D. J. NORMAN.

Cyclic processes involved in the manufacture of sodium nitrate from Chilean caliche. M. A. HAMID (J. Indian Chem. Soc., 1927, 4, 515—524).—The results of the investigation at 25° of the ternary system, water-sodium sulphate-sodium nitrate previously obtained (A., 1926, 245) are applied to the leaching of sodium nitrate from Chile saltpetre. Isothermal addition of water to an equimolecular mixture of sodium nitrate and sodium sulphate at first only results in the formation of darapskite ($\text{Na}_2\text{SO}_4 \cdot \text{NaNO}_3 \cdot \text{H}_2\text{O}$) until the whole solid phase consists of this compound. Further addition of water causes dissociation of this compound, the whole of the sodium nitrate so produced passing into solution while the proportion of sodium sulphate in the solid phase increases until the darapskite is completely dissociated. Throughout these changes the composition of the solution remains constant (c) and represents the most concentrated solution of sodium nitrate which can be obtained, still further addition of water causing the composition of the solution to change in the direction of diminishing sodium nitrate content. The same solution is the most concentrated, with respect to sodium nitrate, which can be obtained when water is added to a mixture in which the weight of sodium nitrate present is less than 60% of that of the sodium sulphate present (number of mols. of sodium sulphate is greater than the number of mols. of sodium nitrate). When the number of mols. of sodium nitrate in the dry mixture exceeds the number of mols. of sodium sulphate by x , it is possible to obtain a quantity of a solution (d) of constant composition

(containing sodium nitrate and darapskite) corresponding to x , whilst the solid phase consists of pure darapskite which, if separated from the solution (d), and again treated with water, yields the solution (c) of constant composition. The phase reactions involved in the Guggenheim leaching process (cf. B.P. 188,634 and 192,032; B., 1924, 94), which involves the system water-sodium sulphate-sodium nitrate-magnesium sulphate-magnesium nitrate, are discussed, and it is shown that by maintaining a sufficient quantity of magnesium salts in the leaching solution and in the raw material the whole of the sodium sulphate-nitrate compound can be decomposed, the sodium sulphate combining with the magnesium sulphate to form astrakhanite ($\text{Na}_2\text{SO}_4 \cdot \text{MgSO}_4 \cdot 4\text{H}_2\text{O}$). Cooling the solution so obtained to a temperature not sufficiently low to cause the separation of the magnesium nitrate present will cause the precipitation of practically pure sodium nitrate.

J. W. BAKER.

Reduction of sodium sulphate to sodium sulphide, particularly by hydrogen and carbon monoxide in the presence of catalysts. P. P. BUDNIKOV and E. SHILOV (J.S.C.I., 1928, 47, 111—113 r).—Sodium sulphate was not reduced by carbon monoxide in the absence of catalysts, at least below 850°. In the presence of carbon reduction was rapid, and it is considered that the carbon acts catalytically and not as a direct reducing agent. Nickel chloride aided the reduction by carbon monoxide, but to a much less extent than carbon. In contradistinction to carbon monoxide, sodium sulphate was reduced by hydrogen in the absence of a catalyst, the reaction beginning at about 700° and increasing rapidly accompanied by secondary decomposition reactions at higher temperatures. The reaction is endothermic. Nickel and nickel compounds exercised a catalytic action on the reduction and also on the decomposition of the reduction products, the yield of sulphide at temperatures above 900° being less in the presence of the catalyst than without it. Loss of sodium sulphide occurred through interaction with sodium sulphate at 1000° or higher. (Cf. B., 1928, 446.)

C. A. KING.

Explosion and fire risks with potassium and ammonium persulphates, and the most suitable methods of packing and storage. G. AGDE and E. ALBERTI (Chem.-Ztg., 1928, 52, 229—232).—To study the extent of possible risk of explosion or firing of these salts, they were heated at a steady rate and the gas evolution was measured at regular intervals. It was found that neither the pure persulphates nor mixtures containing 10% of organic impurity would explode or cause rapid combustion of the organic matter, which was simply charred. The amount of gas evolved at any particular temperature was always greater in the presence of impurity, and the ammonium salt was less stable than that of potassium. Iron cases, with asbestos covering, are therefore considered unnecessary for packing, and wooden boxes of which the surface has been coated with paraffin are recommended.

R. H. GRIFFITH.

Chemistry and technology of zirconium oxide. H. TRAPP (Chem.-Ztg., 1928, 52, 365—367).—In the preparation of zirconia from zircon, the mineral is decomposed by fusion with an alkali and, after leaching and

washing, the residue consists of hydrated zirconia containing after ignition 90% ZrO_2 . The moist residue may be converted into the pure oxide by stirring it in paste form with ammonium hydrogen oxalate, removing the insoluble material, and crystallising out the ammonium zirconyloxalate. This compound forms large, dense crystals which yield pure zirconia on ignition in air. Oxide ores, *e.g.*, zirkite, are decomposed by fusion with acid sulphates or by heating with concentrated sulphuric acid, and from the solution of zirconium sulphate so obtained zirconium phosphate is precipitated by addition of sodium hydrogen phosphate. The precipitate is washed and dissolved in ammonium hydrogen oxalate solution, and the ammonium phosphate formed is separated by crystallisation from the ammonium zirconyloxalate, which is then recovered by further evaporation and purified by recrystallisation. An alternative treatment of the sulphate solution comprises evaporation and partial neutralisation to obtain a liquor containing 8–10% ZrO_2 which is treated with potassium sulphate and oxalic acid. The resulting potassium zirconyloxalate is separated from the mother-liquor, purified by recrystallisation from hot water, and decomposed with alkali carbonate or hydroxide. For the recovery of zirconia from baddeleyite ores on a large scale some modification of the basic sulphate process appears to be the only economical procedure. A. R. POWELL.

Crushing and grinding. I. Surface measurement of quartz particles. J. GROSS and S. R. ZIMMERLEY (Amer. Inst. Min. Met. Tech. Pub., 1928, 46, 16 pp.).—Crushed quartz, free from iron, was agitated with 3.66*N*-hydrofluoric acid at constant temperature for 30 min. or longer, the residual particles then being ignited and weighed. The initial rate of dissolution was determined by the expression $(A + at)/(1 + kt)$, where A is the initial rate, t is the time in hrs., and a and k are constants. Three different specimens had the same rate of dissolution; a quartz crystal does not, however, dissolve equally rapidly in all directions. CHEMICAL ABSTRACTS.

Rates of absorption of sulphurous gases by alkaline solutions. P. RIOU and P. A. BÉRARD (Compt. rend., 1928, 186, 1433–1436).—The rate of absorption of the vapours produced by the combustion of sulphur by milk of lime and by solutions of lime water of various concentrations, increases from the moment of separation of solid matter at a rate proportional to the concentration of the latter, and decreases gradually with rise in the temperature. J. GRANT.

Determination of sulphur dioxide in small amounts in the atmosphere. R. J. MCKAY and D. E. ACKERMAN (Ind. Eng. Chem., 1928, 20, 538–542).—The method described is suitable for the determination of amounts of sulphur dioxide varying from 0.05 to 20 parts per million, and has been thoroughly checked on synthetic mixtures. The sample of air is drawn by an evacuation method into a large bottle containing a dilute starch-iodine mixture, which is then withdrawn and brought to the same intensity of blue as a blank by addition of a dilute standard solution of iodine. The apparatus, method, and the essential precautions are described in considerable detail. With alteration in

the size of sample and strength of solutions the method is applicable to the determination of sulphur dioxide in flue gases. If hydrogen sulphide is present in appreciable amounts, it must be determined separately and the necessary correction made. J. S. CARTER.

Catalytic refining of bromine. P. T. DANILITSCHENKO and M. RAVITSCH (J. Russ. Phys. Chem. Soc., 1927, 59, 953–968).—Bromine is passed over heated activated charcoal and iron oxide, which act as catalysts and absorb organic impurities. By this method at a temperature of 115° bromine can be practically completely freed from chlorine. A. RATCLIFFE.

Analysis of bleach liquors etc. HINTZMANN.—See VI. **Chrome alum liquors.** SCHINDLER and KLANFER.—See XV. **Limestones for Bordeaux mixtures.** CARR and BEMILLER.—See XVI. **Silver halide emulsions.** SHEPPARD and CROUCH.—See XXI. **Effluents from potash works.** EMMERLING.—See XXIII.

PATENTS.

Separation of ammonia from gases and mixtures containing it.—F. UHDE (B.P. 272,929, 15.6.27. Ger., 15.6.26).—Liquid ammonia separated from the gaseous mixture by cooling is evaporated under low pressure in a heat-exchanger and led in counter-current to the cooled gas mixture to cool it further and obtain more ammonia. With a mixture containing too small an amount of ammonia to obtain the requisite low temperature, a portion of the evaporated ammonia is compressed and added to the mixture to be cooled.

W. G. CAREY.

Ammonia condenser. S. O. LOKEY (U.S.P. 1,669,943, 15.5.28. Appl., 6.11.26).—The cooling medium is circulated through conduits in the still, and the product to be condensed is mixed with condensate and sprayed against the cooling surfaces. C. O. HARVEY.

Manufacture of ammonium chloride crystals. J. W. MOORE, W. G. POLACK, and CASTNER-KELLNER ALKALI Co., LTD. (B.P. 290,045, 18.5.27. Addn. to B.P. 273,093; B., 1927, 652).—In the interaction of synthetic ammonia and gaseous hydrochloric acid at 230–310°, gases saturated with moisture at ordinary temperature may be used, the heat of reaction preventing condensation of moisture. W. G. CAREY.

Production of aluminium oxide or products containing it from material containing aluminium sulphide. T. R. HAGLUND (B.P. 289,639, 3.5.27).—Slags containing aluminium sulphide are treated with air, sulphur dioxide, or other oxidising agent in such proportions as to liberate in the elementary state the greater part of the sulphur content at such a temperature that the liberated sulphur is distilled out of the mass. The addition of a small quantity of steam to the oxidising gas accelerates the reaction, which may be further hastened by introducing hydrogen chloride together with the steam. The treatment with sulphur dioxide may also be carried out in an aqueous suspension of the finely-divided slag either with or without heat and pressure; in this case the reaction product is aluminium hydroxide if a deficiency of sulphur dioxide is used, and aluminium sulphite if an excess is used. When carbon dioxide or carbon monoxide is employed as the oxidising

agent at 800—900°; the reaction products contain carbonyl sulphide and carbon disulphide.

A. R. POWELL.

Manufacture of titanium compounds. H. WADE. From TITAN Co. A./S. (B.P. 289,111, 17.11.26).—The precipitate of metatitanic acid or of a basic titanium salt obtained by hydrolysis of solutions of quadrivalent titanium at elevated temperatures is rendered soluble in dilute mineral acids and in concentrated solutions of organic acids by digesting it at 150° with a concentrated solution of an alkali hydroxide, whereby the titanium is converted into a fine crystalline powder of an alkali titanate. This powder dissolves readily in oxalic, lactic, and tartaric acids, and may be used for the manufacture of potassium titanyl oxalate etc.

A. R. POWELL.

Apparatus for production of gaseous ozonides. E. J. BAGNALL (A. M. BAGNALL, extrix.), Assr. to KNOX TERPEZONE Co., INC. (U.S.P. 1,668,884, 8.5.28. Appl., 22.11.22. Renewed 30.7.27).—The apparatus comprises an ozone generator, an evaporator, and a dryer in communication with one another, and means for supplying air to the dryer and for air-cooling the generator.

A. R. POWELL.

Manufacture of diammonium phosphate. R. GRIESSBACH, O. BALZ, and A. RÖSSLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,670,504, 22.5.28. Appl., 19.2.26. Ger., 20.2.25).—See B.P. 256,137; B., 1926, 821.

Manufacture of persalts. F. NOLL (U.S.P. 1,669,997, 15.5.28. Appl., 21.8.23. Ger., 28.8.22).—See B.P. 202,985; B., 1924, 980.

Manufacture of zinc oxide. J. A. SINGMASTER, F. G. BREYER, and E. H. BUNCE, Assrs. to NEW JERSEY ZINC Co. (U.S.P. 1,670,169, 15.5.28. Appl., 27.3.26).—See B.P. 268,301; B., 1927, 916.

Extraction of krypton and xenon from air. A. J. A. BLARINGHEIM, Assr. to SOC. ANON. D'ÉCLAIRAGE ET D'APPLICATIONS ÉLECTRIQUES (U.S.P. 1,670,014, 15.5.28. Appl., 26.5.24. Fr., 29.6.23).—See B.P. 218,266; B., 1925, 242.

Hydrogen from coke-oven gases (B.P. 271,491).—See II. **Recovery of soda ash** (B.P. 288,699).—See V. **Plastic lime** (U.S.P. 1,664,598).—See IX. **Activation of oxygen** (B.P. 265,636).—See XI. **Treatment of phosphates** (B.P. 264,867). **Fertilisers** (B.P. 290,075).—See XVI.

VIII.—GLASS; CERAMICS.

Causes and removal of certain heterogeneities in glass. L. W. TILTON, A. N. FINN, and A. Q. TOOL (U.S. Bur. Standards, Sci. Paper No. 572, 1928, 22, 719—736).—Refractive indices of six well-annealed samples of a barium flint optical glass from the same melt revealed individual deviations from the mean having a total spread from -37×10^{-6} to 26×10^{-6} . Thermal expansion and heat absorption measurements indicated a commencement of the critical temperature zone at about 545°, and a careful re-annealing was then given in which the samples were heated to 560° and cooled at the rate of 80° per day, with a break at 486°, during which

the temperature was held constant for 13 days. The range of the deviations was so reduced to 21×10^{-6} . A second re-annealing with greater precautions to reduce furnace gradients brought this range down to 5×10^{-6} .

A. COUSEN.

[Cause of opacity of] white enamel. R. D. COOKE (J. Amer. Ceram. Soc., 1928, 11, 126—130).—A white enamel is heterogeneous, being an intimate mixture of two or more transparent substances of different indices of refraction. Opaque frits contain crystals of fluorine compounds, such as sodium and calcium fluorides, having low indices of refraction compared with the silicate glass. Whiteness is defined as the ratio of the total light energy received on a surface to the total light energy reflected from it. In order to formulate a satisfactory test of whiteness, a standard enamel, with additions of 0, 4, 8, 12, and 16% of tin oxide, was milled. Sample plates were sprayed with each of these enamels in various thicknesses. It is shown that the reflexion factor increases with increase in the amount of tin oxide and with the thickness. By measuring the thickness and the reflexion factor, the intrinsic whiteness of the enamel can be determined.

A. T. GREEN.

Determination of iron in glass sand. G. E. F. LUNDELL and H. B. KNOWLES (J. Amer. Ceram. Soc., 1928, 11, 119—125).—Methods for determining small quantities of iron in sands are considered and their relative accuracies discussed. Of these, the electrometric titration method, the hydrogen sulphide reduction method, and the gravimetric method are described in detail. The colorimetric procedure gives low and the others high results. The electrometric titration method is both rapid and satisfactory provided platinum is excluded and the blank correction properly carried out.

A. T. GREEN.

Physical properties of artificial aluminous abrasives. R. C. PURDY and A. E. MACGEE (J. Amer. Ceram. Soc., 1928, 11, 192—203).—Test pieces of artificial corundum differently bonded were made by the normal puddled process and turned to a size satisfactory for testing. The firing, which varied from 70 hrs. at cone 12 to 120 hrs. at cone 10, was conducted in commercial kilns at various plants. On testing, it was found that porosity, specific gravity, elasticity, and thermal expansion decrease, whilst resistance to heat shock, impact strength, modulus of rupture, crushing strength, and mechanical endurance increase, with increasing percentages of bond and resulting hardness of grade. Above 300° the expansion of the bond is less than that of the artificial corundum, whilst below this temperature the reverse holds, the expansions of the two constituents tending towards equality. The properties of similarly constituted abrasives vary with variations in plant practice.

A. T. GREEN.

Measurements of the expansion of refractory bricks at 1600°. K. ENDELL and W. SIEGER (Arch. Eisenhüttenw., 1927—8, 1, 721—724; Stahl u. Eisen, 1928, 48, 722).—After a critical discussion of the merits of the methods used for determining the expansion of refractories at high temperatures by previous workers, results obtained by a new method devised by the authors are recorded. Fireclay bricks with a medium or high

content of alumina expand only slightly but evenly over the range 20—1300° and then begin to contract. Fire-clay bricks with a high content of quartz exhibit the usual irregularities in expansion caused by the various transformations which take place on heating quartz. Magnesite bricks expand considerably but evenly up to 1500°, then begin to contract. Silica bricks of d 2.35 retain a constant volume between 600° and 1600°; those having d 2.40 are constant up to 1450°, and those having d 2.45—2.54 expand considerably up to 1250° and at 1600° their total linear expansion is 2.85—3.35% of the original length. A. R. POWELL.

PATENTS.

Pressing of articles of fused silica. QUARTZ & SILICE (B.P. 283,174, 28.12.27. Fr., 6.1.27).—Blocks of silica are heated electrically on a furnace bed of "zirconium" or graphite at 2200° to cause volatilisation of silica and so prevent adherence to the bed; the mass is then rapidly pressed in moulds heated at 1000° and annealed in a closed chamber heated at 1200°.

W. G. CAREY.

Manufacture of refractory compositions. J. S. WITHERS. From VITREFRAN Co. (B.P. 289,560, 1.2.27).—Material containing about 72% Al_2O_3 and 28% SiO_2 , and resembling mullite, is prepared by fusing suitable quantities of a mixture of precipitated alumina and material such as cyanite, which is preferably concentrated to about 95% purity. The charge is fused in a three-phase electrical furnace under such conditions that the bath is kept in a fluid condition so that heavy impurities sink to the bottom and, like the crust at the top, can subsequently be stripped from the product. A more compact material suitable for lining furnaces containing glasses, enamels, etc. contains up to 3% K_2O and 0.25% P_2O_5 , but not more than 1% CaO and MgO . L. A. COLES.

[Soda-lime] glass. B. LONG, Assr. to SOC. ANON. DES MANUF. DES GLACES & PROD. CHIM. DE ST.-GOBAIN, CHAUNY, & CIREY (U.S.P. 1,669,908, 15.5.28. Appl., 12.1.26).—See B.P. 264,490; B., 1927, 602.

Annealing of glass articles. UNITED GLASS BOTTLE MANUFRS., LTD., E. A. C. PRYOR, and F. A. HURLBUT (B.P. 289,501, 29.10.26 and 26.8.27).

IX.—BUILDING MATERIALS.

Action of sulphates on the components of Portland cement. T. THORVALDSON, V. A. VIGFUSSON, and R. K. LARMOUR (Trans. Roy. Soc. Canada, 1927, [iii], 21, III, 295—310).—The effect of sulphate solutions on cement may be observed by following the expansion of a bar of cement mortar, and the decrease in tensile strength with time, when immersed in the sulphate solution. In this way the action of sulphates on tricalcium silicate, β -dicalcium silicate, and tricalcium aluminate, which, according to Shepherd, Rankin, and Wright (A., 1911, ii, 725; 1915, ii, 50), are the chief constituents of normal Portland cement, has been studied. Mortars made from pure tricalcium silicate and pure β -dicalcium silicate are not disintegrated in solutions of sodium sulphate, but expand and are disintegrated in solutions of magnesium sulphate. If tricalcium aluminate is added to either, the resulting

mortar disintegrates rapidly in solutions both of sodium and of magnesium sulphate. A mortar made from a composite cement containing 21.5% of tricalcium aluminate, 52.7% of tricalcium silicate, and 25.8% of β -dicalcium silicate mixed with Ottawa sand in the proportion of 1 : 7, has the usual tensile strength of a similar mortar made from normal Portland cement, and is affected by solutions of sodium, magnesium, and calcium sulphates in exactly the same way. This is strong evidence in favour of Shepherd, Rankin, and Wright's views (*loc. cit.*) as to the composition of normal Portland cement clinker. Steam treatment has been found to increase enormously the resistance of cement mortar to the disintegrating action of sulphate solutions.

M. S. BURR.

Deterioration of concrete by corrosive waters. R. GRÜN (Chem. Fabr., 1928, 281—283, 294—295).—The most important damage is caused by sulphuric acid either combined in sea-water or free in natural acid water. Ordinary concrete can be made resistant by producing a dense non-porous aggregate of uniformly sized material. At least 400 kg. of cement/m.³ must be used. Painting with bitumen is beneficial and better than covering with clinker and cement. Aluminous cement with 7—50% Al_2O_3 is very resistant to magnesium sulphate, but its behaviour in presence of sodium sulphate is doubtful. The resistance of pozzuolana to corrosion is very great, as is shown by the existence of water mains in good condition to-day constructed by the Romans of this material. The effect of its addition to concrete is to increase the density and to liberate lime, which acts protectively. It can only be used with slow-setting concrete. Cements prepared from blast-furnace slag fall into the same class; or a mixture of blast-furnace slag and Portland cement clinker may be used. Those made from acid slags have not a high initial strength, but if the clinker content is low they are very resistant to salt water. Aluminous slags have the best "hydraulic" qualities of any, but are not so resistant to corrosion as might be expected. Basic and magnesian slags also make good cement, but it is not resistant to salt water. Generally speaking, all these cements have less initial strength than Portland cement, but greater resistance to corrosion.

C. IRWIN.

Examination of cements and plastic masses, with especial reference to their behaviour in electrotechnique. I. Binding power. W. NAGEL and J. GRÜSS (Wiss. Veröff. Siemens-Konz., 1928, 6, [2], 150—173).—The binding power of a cement or plastic mass is defined as the weight in kg. required to pull a metal rod 8 mm. in diam. from a hole, 10 mm. deep and 10 mm. in diam., in which the rod is embedded in a porcelain plate by means of the cement under test. For zinc oxychloride cement with borax to retard setting, maximum binding power is obtained with $4ZnO : 1ZnCl_2$ when the zinc oxide used has been well burnt. For magnesium oxychloride cement with or without a filler the maximum binding power is obtained with $4MgO : 1MgCl_2$. The greatest strength is obtained with sand as a filler; calcite is almost as good, but kaolin is appreciably inferior. The quantity of alum used in mixing Keene's cement has little effect on its binding

power, but with 10% a slight maximum occurs in the curve. The binding power of artificial resins such as "Glyptal" or coumarone resin increases roughly in proportion to the amount of filler used; natural colophony behaves somewhat similarly.

A. R. POWELL.

Effect of decay on the chemical composition of wood. L. F. HAWLEY, L. C. FLECK, and C. A. RICHARDS (Ind. Eng. Chem., 1928, 20, 504—507).—Analyses of the sapwood of two hard woods and two soft woods before and after attack by a white-rot fungus (*Polystictus hirsutus*) and a brown-rot fungus (*Lenzites striata*) indicate that the pentosans in the cellulose are attacked more rapidly in proportion to the amount present than are the hexosans; also that the more readily hydrolysed constituents of the cellulose, as determined by the hydrolysis number (cf. B., 1927, 598), are, in general, more rapidly attacked than is the more stable cellulose, though the two processes appear to start simultaneously. In the early stages of decay the action of the white-rot fungus, *P. hirsutus*, resembles that of the brown-rot fungi in attacking preferentially the cellulose component of the lignocellulose complex.

D. J. NORMAN.

PATENTS.

Manufacture of cement. H. O. MOEBIUS, Assr. to AMER. MIAG CORP. (U.S.P. 1,680,269, 15.5.28. Appl., 2.8.27).—The pulverised raw material is burnt to clinkers, then partly cooled, crushed, and agitated in a current of cool air.

H. ROYAL-DAWSON.

Manufacture of coloured Portland cement. AMME-LUTHER WERKE BRAUNSCHWEIG DER "MIAG" MÜHLENBAU & IND. A.-G. (B.P. 284,295, 27.1.28. Ger., 27.1.27).—A metal pigment, e.g., an oxide of chromium, nickel, cobalt, or copper, and a salt capable of forming a coloured melt with it on calcination, e.g., a phosphate, borate, or halide, are added to raw materials used for making Portland cement. Reducing agents are also added if the raw material contains iron oxide, so that this is reduced and can be removed. L. A. COLES.

Cementitious material. H. E. BROOKBY and C. K. ROOS, Assrs. to U.S. GYPSUM CORP. (U.S.P. 1,668,548, 8.5.28. Appl., 23.4.24).—The material consists of powdered raw calcium sulphate blended, without calcination, with sodium thiosulphate. H. ROYAL-DAWSON.

[Production of cellular] concrete. J. A. THOMPSON (B.P. 272,479, 24.5.27. Austral., 12.6.26).—Concrete containing material for the generation of gases during setting so as to produce a cellular mass is allowed to set in hermetically sealed moulds in which the pressure is reduced to about $14\frac{1}{2}$ — $10\frac{1}{2}$ lb./in.², according to the depth or thickness of the mass.

L. A. COLES.

Manufacture of plastic hydrated lime. H. DITTLINGER (U.S.P. 1,664,598, 3.4.28. Appl., 25.6.26).—Hydrated lime that gives a "putty" of the requisite plasticity immediately on mixing with water, and does not therefore require seasoning, is made by shattering the dry hydrate by causing it to impinge upon a beater rotating at high speed within a casing. The hydrate may be prepared by using a dilute aqueous sodium thiosulphate solution for slaking, in order further to increase the plasticity of the product. F. G. CLARKE.

Transformation of slag-forming waste materials (such as domestic or industrial refuse, gutter residues, etc.) into slag-sand of various grain-size. MUSAG GES. F. DEN BAU VON MÜLL- U. SCHLACKEN-VERWERTUNGSANLAGEN, A.-G., and A. GROTE (B.P. 280,902, 9.11.27. Ger., 20.11.26).—The waste materials are burned in the front portion of an obliquely mounted rotary furnace, and the resultant slag is superheated in the remaining portion of the furnace in such a way that the softened lumps of slag are broken up and granulated.

C. JEPSON.

Wood preservation. H. W. WALKER (U.S.P. 1,670,086, 15.5.28. Appl., 25.7.24. Renewed 12.4.27).—The wood is impregnated with the product of a water-soluble dye and an organic constituent of the wood which is toxic to wood-boring organisms and has a solubility in cold water slightly in excess of the minimum toxic concentration.

H. ROYAL-DAWSON.

Bituminous coating compositions. G. A. WELLINGS and E. JOHNSTONE (B.P. 289,737, 1.11.26).—A solution of bitumen containing gold size and a solution or suspension of sulphur with or without a filter.

L. A. COLES.

Manufacture of asbestos bodies. H. GERDIEN, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,670,659, 22.5.28. Appl., 7.11.25. Ger., 17.10.24).—See B.P. 241,576; B., 1927, 389.

[Press for] the manufacture of bricks. H. ACKERMANN, and SCHEIDHAUER & GIESSING A.-G. (B.P. 267,949, 17.3.27. Ger., 17.3.26).

Production of slabs for walls, partition walls, etc. A. P. LAURIE (B.P. 290,042, 12.5.27).

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Rotating tube drying furnace. J. A. L. ORTLEPP (J. Chem. Met. Soc. S. Africa, 1928, 28, 210—213).—A rotary drying furnace particularly designed for drying tin concentrates out of contact with flue dust and excessive draughts consists of a steel tube 8 ft. long and 8 in. in diam. inclined at a slope of 1 in 12. The tube is heated externally, excess air being admitted to prevent burning of the metal. Material to be dried is fed from a top drying plate into a hopper to the tube, from which it is discharged through a suitable screen into a box, which also regulates the current of air and steam through the drying tube.

C. A. KING.

Smelting titaniferous ores of iron. A. STANSFIELD (Trans. Canad. Inst. Min. and Met., 1927, 30, 802—818).—Titaniferous iron ores with a high percentage of titania can be smelted in blast furnaces under suitable conditions to obtain a pig iron and a fusible slag containing about 20% of titanium oxide in the form of a lower oxide, probably Ti_2O_3 . Under too highly oxidising conditions no reduction of the titania takes place and pasty slags are formed, whereas under very highly reducing conditions titanium carbide and cyanonitride are obtained and the slags are fusible only at very high temperatures.

A. R. POWELL.

Phosphorus and arsenic in steel and the substitution theory. A. E. CAMERON (Trans. Canad. Inst. Min. and Met., 1927, 30, 858—868).—The solid solubility of phosphorus and of carbon in γ -iron is about 1.7%, and the effects of the two elements on the mechanical properties of iron are approximately the same. Carbon, however, increases the tensile strength owing to the insolubility of the carbide in α -iron and its separation on cooling together with the resistance to slip afforded by the finely-crystalline state of the eutectoid, whereas phosphorus can cause hardening only by producing distortion in the lattice of α -iron. This distortion of the body-centred lattice of α -iron produces the well-known cold-shortness of phosphoretic steels. Most steels contain arsenic up to about 0.1%, but as the solid solubility of arsenic in iron is 6.8% the hardening and, therefore, the embrittling effect of arsenic on steel is much less marked than that of phosphorus. An embrittling effect may, however, be caused by segregation of the arsenic due to the presence of carbon in solid solution producing areas in which the arsenic content is very much higher than the average. The metal in these areas would then have a lower m.p. than the remainder, and during heat-treatment may be in a semi-plastic state, thus causing serious weakness and possibly breakdown during forging.

A. R. POWELL.

Behaviour of basic open-hearth steel sheets during rolling. E. POHL (Stahl u. Eisen, 1928, 48, 649—656).—The mechanical properties of a basic open-hearth mild steel having a tensile strength at 20° of 49 kg./mm.² have been determined between 20° and 500°, and the effect on these properties of a tensile strain above and below the elastic limit has been ascertained. A strain below the elastic limit has no effect on the mechanical properties, but a strain above that limit induces an increase in hardness and tensile strength, but a decrease in impact resistance, especially at temperatures between 200° and 300°. It is recommended, therefore, that steel boilers should be constructed to work at temperatures above 350° so as to avoid the range of "blue brittleness" between 200° and 300°.

A. R. POWELL.

[Mechanical properties of] boiler-plate [steels]. A. POMP (Stahl u. Eisen, 1928, 48, 681—689).—The effects of temperature, ageing, and recrystallisation on the mechanical properties of 14 samples of boiler-plate steels have been determined and are recorded graphically. All the samples tested contained 0.05—0.2% Cu, 0.02—0.04% S, 0.02—0.04% P, 0.4—0.6% Mn, and 0—0.2% Si. The carbon content in the plain steels was 0.05, 0.15, or 0.25%, and in the nickel steels the effect of 3% and 5% of nickel was determined. The elastic limit, limit of proportionality, and permanent strength decreased in all cases with rise of temperature, the actual values depending on the carbon, manganese, and nickel content. The tensile strength increased to a maximum between 200° and 400°, the elongation and reduction of area decreased to a minimum at 100—200° and then rose sharply, and the impact resistance rose abruptly to a maximum at 50—150° and then fell rapidly. Prolonged ageing of the worked plates at 200° resulted in a slight increase in the tensile strength and elastic limit with a corresponding decrease in the elongation; the

impact strength of the mild steel plates was considerably decreased after this treatment, and that of the steel plates containing 0.25% C fell appreciably, whereas the nickel steel plates showed only a slight decrease in strength. Annealing just below the A3 point caused a small decrease in the tensile strength and a large decrease in the impact strength of the plain carbon steels, but a slight increase in both these values of the nickel steels. The superiority of the latter for boiler plates is thus established.

A. R. POWELL.

Impact resistance of steels at low temperatures. J. F. MORRISON and A. E. CAMERON (Trans. Canad. Inst. Min. and Met., 1927, 30, 839—857).—The impact resistance of hypoeutectoidal steels is decreased by exposure to low temperatures. At —30° the impact resistance of a normalised steel containing 0.1% C is about one tenth that of the same steel at 20°, whilst a normalised steel with 0.34% C has an impact strength at 20° three times as great as at —30°. The same steels in the quenched and fully tempered condition have an impact strength at —30° which is only slightly lower than that at 20°. For structural purposes in cities where a low winter temperature is experienced, the best resistance to shock is obtained by quenching from 900° followed by tempering at 610°.

A. R. POWELL.

Determination of oxides in steel. F. WILLEMS (Arch. Eisenhüttenw., 1927—8, 1, 605—608; Stahl u. Eisen, 1928, 48, 623—624).—Manganous oxide may be satisfactorily determined in steel by shaking fine turnings or filings of the metal with a solution of iodine in absolute alcohol. The operation is carried out in a closed flask from which the air has been completely displaced by nitrogen, and, when dissolution is complete, the residue is collected on a "Cella" filter, washed with absolute alcohol, and dissolved in hydrochloric acid. Manganese is determined in this solution by any of the ordinary methods. Silica and alumina inclusions in steel may also be satisfactorily determined by this process, but the copper ammonium chloride and the bromine methods of Oberhoffer and Ammann (B., 1927, 967) give equally satisfactory results for these constituents, and are more easily carried out.

A. R. POWELL.

Precious metals in the Sudbury [nickel] ores and their recovery. C. LANGER, SEN., and C. JOHNSON (Trans. Canad. Inst. Min. and Met., 1927, 30, 903—908).—The residues from the carbonyl volatilisation of nickel in the Mond process of treating copper-nickel sulphide ores contain practically the whole of the precious metals contained in the original ore and represent a concentration of these metals of 16,000:1. A typical analysis of these residues is 1.85% Pt, 1.91% Pd, 0.56% Au, 0.39% Ir, Ru, and Rh, and 15.42% Ag, together with antimony, lead, nickel, copper, arsenic, sulphur, selenium, and tellurium. The residues are roasted to expel volatile constituents, and smelted with litharge and alkaline fluxes to obtain a lead regulus. This is cupelled with more silver to give at least six times as much silver as other precious metals in the resulting bullion, which is then electrolysed in a silver nitrate bath. Pure silver is thus obtained at the cathode and a rich slime at the anode, from which the

platinum metals and gold may be recovered by the usual procedure.

A. R. POWELL.

Recrystallisation of α -brass. A. BASS and R. GLOCKER (*Z. Metallk.*, 1928, **20**, 179—183).—The temperature at which 70:30 brass commences to recrystallise is dependent on the nature of the deformation to which the metal has been subjected, but the size of the new crystals and their orientation are not affected by the nature of the previous work. The size of the new crystals is greater the higher the annealing temperature and the smaller the degree of deformation, and their orientation, as determined by X-ray examination, is similar to that of annealed silver after severe deformation. The presence of lead up to 1.2% has no influence on the tensile strength, elongation, or grain size after annealing, but a very small iron content suffices to restrain the grain growth very considerably below 700°. Above this temperature iron causes a rapid increase in grain size with further rise in the annealing temperature. With 63:37 brass the presence of β -grains may considerably affect the shape of the recrystallisation diagram.

A. R. POWELL.

Industrial control of cold-hardening and annealing [of brasses] by light-load ball tests. P. NICOLAU (*Rev. Mét.*, 1928, **25**, 155—168).—A discussion of tests suitable for the industrial control of low-temperature annealing of brass cartridge case collars after deformation, in order to prevent season cracking. The mercury or mercuric salt test and microscopical examination are impracticable, but a Brinell hardness test with a 1.58 mm. ball under a load of 10 kg. applied for 15 sec. gives consistent results, and the method is suitable for unskilled labour. A suitable apparatus is described, and results for 67/33 and 72/28 brasses are discussed in detail. In each case the strain is relieved by annealing at temperatures between 300° and 350°, but between 350° and 400° the hardness curves show a point of inflexion not previously noticed. Severe working lowers the temperature at which strain is removed by low-temperature annealing, but increases the final hardness of the annealed metal.

W. HUME-ROTHERY.

Structure of brass tubes. Surface faults in bent tubes and their prevention. F. OSTERMANN (*Z. Metallk.*, 1928, **20**, 186—188).—Some characteristic crystal structures of the surface of extruded and of cold-drawn and annealed brass tubes are illustrated by photomicrographs. The copper content of brass for extruding tubes should not be below 61.3%, and the extrusion temperature must not exceed 600° if satisfactory bends are to be obtained. A higher temperature leads to the formation of coarse needle-shaped crystals which cause a rough, hackly surface, or even fracture, to be produced during bending.

A. R. POWELL.

Metallurgy of ancient bronzes. D. BUTEȘCU (*Bûl. Soc. Romane Stiin.*, 1928, **30**, 83—98).—The metal of a number of bronze implements found at Drăjna-de-Jos, Rumania, and probably dating from 1100—1400 B.C., has been analysed. In addition to copper and tin, small amounts of iron, manganese, and lead were found. There was some evidence that rapid quenching had been employed to secure hardness.

R. CUTHILL.

Aluminium alloys in motor construction. H. STEUDEL (*Z. Metallk.*, 1928, **20**, 165—178).—A review of the preparation, properties, and uses of various age-hardening aluminium alloys showing how advantage is taken of these properties in the construction of cylinders, piston rods, and other parts of motors, especially for aviation purposes.

A. R. POWELL.

Extraction of vanadium and its salts from residues containing vanadium. N. N. EFREMOV and A. ROSENBERG (*J. Russ. Phys. Chem. Soc.*, 1927, **59**, 689—699).—The powdered dry waste residue from the working of vanadium ores is treated with boiling nitric acid and heated on the water-bath for 2 hrs. The precipitate thus formed consists mainly of vanadic oxide, and is dissolved in boiling sodium hydroxide and converted into sodium metavanadate. The contaminations present remain undissolved and are removed by filtration. The filtrate contains sodium vanadate, sodium aluminate, and traces of sodium silicate. The aluminate is removed by acidifying with nitric acid and subsequent boiling with a strong solution of sodium carbonate. The precipitate formed is collected, the filtrate made faintly acid with nitric acid, boiled to drive off carbon dioxide, and precipitated with an excess of ammonia solution. The filtrate, containing pure sodium metavanadate, is boiled and ammonium metavanadate precipitated with an excess of a hot saturated ammonium chloride solution. Uranium and further quantities of ammonium vanadate are recovered from the first filtrate. Vanadic acid precipitated on asbestos fibre is determined by treating the asbestos with a solution of sodium hydroxide, hot water, and hydrogen peroxide, filtering, acidifying the filtrate with sulphuric acid, reducing vanadic acid to a vanadyl salt with sulphur dioxide, boiling in a stream of carbon dioxide to remove excess of sulphur dioxide, and titrating with permanganate.

A. RATCLIFFE.

Orientation of the crystallites of rolled and drawn metals. Crystallographic significance of the mechanism of slip during drawing, pressing, and rolling of metals. G. TAMMANN and A. HEINZEL (*Arch. Eisenhüttenw.*, 1927—8, **1**, 663—667; *Stahl u. Eisen*, 1928, **48**, 693—694).—In metals with a face-centred cubic lattice (*e.g.*, silver, copper, gold, and aluminium) slip can take place along four octahedral planes, whereas in metals with a body-centred cubic lattice it may take place along twelve eicositrahedral planes in a direction parallel to the line bisecting the obtuse angle of the eicositrahedral face. In drawing, slip takes place first along that plane which contains the largest component of the tractive force; friction then causes this plane to rotate, and slip ensues along the second plane and so on until eventually all the slip planes are oriented in the direction of the pull. It follows that, under the action of tractive forces, the maximum thrust occurs along the slip planes of hard-drawn metals when a threefold axis of symmetry in the case of face-centred metals, and a twofold axis in the case of body-centred metals, falls in the direction of drawing; the converse is true for compressive forces. The effect of rolling differs from that of drawing or hammering in that after a certain degree of reduction a complete re-orientation of the crystallites takes place;

in the first stage of rolling the change in the orientation is similar to that caused by drawing, and in the second stage it is similar to that caused by hammering. For hard-rolled metals, therefore, the orientation of the crystallites in the plane of rolling is a combination of the effects produced by drawing and hammering.

A. R. POWELL.

Electrolytic copper. W. KÖSTER (Z. Metallk., 1928, 20, 189–191).—When hard-rolled electrolytic copper is annealed the hardness falls more or less proportionately with the rise in temperature up to 500°, which temperature is necessary for complete annealing even with only 3% deformation. If electrolytic copper is subjected to a heat treatment which causes recrystallisation to take place previous to rolling, the reduction of hardness during subsequent annealing takes place within a range of 25–50° at temperatures varying from about 100°, after 93% reduction in thickness, to about 350° for a 3% reduction. This difference in behaviour is ascribed to the elimination of occluded hydrogen during recrystallisation, the copper used in the tests having given up 0.00026% when heated *in vacuo* at 500°. Fire-refined copper does not alter in sp. gr. after cold rolling and annealing, but the value of *d* for electrolytic copper decreases from 8.92 after annealing above 600° to 8.6 after annealing at 1000°. Prolonged annealing at 780° causes a gradual reduction in *d* to 8.42 after 70 hrs. These changes in volume are due to the liberation of hydrogen along the grain boundaries with the consequent formation of fine pores which grow in size with the temperature and time of annealing.

A. R. POWELL.

Potentiometric analysis of binary, ternary, and quaternary tin alloys. H. BRINTZINGER and F. RODIS (Z. Elektrochem., 1928, 34, 246–250).—The reduction of the solutions of tin-antimony alloys by chromous chloride solution has been studied potentiometrically, as far as the production of metallic antimony. Simultaneous separation and determination of the two metals may be effected by converting the sodium thioantimonate and thiostannate, present in the solution after removal of copper and lead, into antimony pentachloride and stannic chloride, respectively, by addition of hydrogen peroxide and hydrochloric acid. The simultaneous separation and determination of copper and tin requires the presence of considerable calcium chloride and of hydrochloric acid in the solution; dissolved oxygen is removed by passing a current of carbon dioxide through the hot solution, the titration being conducted at 90–100°. The first break in the titration curve indicates the reduction from the cupric to the cuprous state, and is easily determined; the second break, corresponding to the reduction from stannic to stannous tin, can only be obtained accurately by allowing a 3 min. interval between the addition of chromous chloride and the potential reading. The two breaks in the bismuth-tin titration curves are due to the reduction first from stannic to stannous and then of the tervalent bismuth ion to metal, whilst those of the iron-tin curve are caused by reduction first of iron and then of tin. Iron, tin, and bismuth, and copper, tin, and bismuth in ternary mixtures are reduced in the order named. The same holds for antimony, copper, and tin mixtures, but owing to the close proximity of

the first two breaks, a fourth break, corresponding to the reduction of tervalent antimony to metal, may be employed. The presence of lead in antimony-copper-tin alloys does not influence the titration, and the amount present may be determined by difference.

H. F. GILLBE.

Application of electrical resistance measurements to the study of atmospheric corrosion of metals. J. C. HUDSON (Proc. Physical Soc., 1928, 40, 107–131).—A description is given of experimental procedure for quantitative field tests on the atmospheric corrosion of metals, based on the determination of the change in resistance of wire specimens produced on exposure. The resistance changes produced in duplicate specimens agree to within 5%. The results obtained are in agreement with those given by determinations of change in weight. For copper the percentage change in resistance was inversely proportional to the wire diameter, which makes it possible to obtain results with thin wires in a few weeks.

C. J. SMITHELLS.

Sulphuric acid. DE BLOIS; also ROBSON.—See VII.

PATENTS.

Rabble furnace. A. N. JETTE (U.S.P. 1,670,038, 15.5.27. Appl., 8.9.26).—The bricks, composing an insulating mantle surrounding a central shaft and carried by supporting rings thereon, are kept in position by vertically-disposed reinforcing rods passing through the mantle and engaging the rings. J. S. G. THOMAS.

Froth-flotation concentration of ores. C. P. LEWIS, ASSR. to MINERALS SEPARATION N. AMER. CORP. (U.S.P. 1,668,917, 8.5.28. Appl., 28.7.25).—The pulp of the oxidised ore is agitated with a xanthate and a soluble silicate under conditions to produce a mineral-bearing froth, which is then separated. H. ROYAL-DAWSON.

Refining of iron. I. G. FARBERIND. A.-G. (B.P. 268,770, 26.3.27. Ger., 1.4.26).—Carbon, sulphur, and phosphorus may be almost completely removed by melting steel scrap with iron oxide obtained by the oxidation of iron carbonyl as described in B.P. 258,313 (B., 1926, 979).

A. R. POWELL.

Production of non-ferrous coated billets of steel or the like. J. M. ROTH (B.P. 290,112, 7.11.27).—Steel billets are faced, coated with a flux, and electrically heated while centred in a mould, into which the coating metal is poured. The composite billet may then be rolled, drawn, or otherwise reduced to rods or wire.

C. A. KING.

Proofing of iron and steel against rust. W. H. COLE (B.P. 289,906, 1.1.27).—A corrosion-resisting deposit is formed by treating iron or steel with a solution containing phosphates of aluminium, zinc, iron, and chromium, to which solution may be added "flux" salts, *e.g.*, diammonium phosphate, sodium diborate, or potassium dichromate.

C. A. KING.

Alloy steel. F. H. ROGERS. From REMY-STAHLEWERKE STAHLSCHEIDT & Co., G.M.B.H. (B.P. 289,990, 28.2.27).—A high-speed tool steel contains 17% W, 7% Ta, 5% Cr, 1.5% V, and 0.9% C, and, if desired, 2% Mo.

F. G. CROSSE.

Treatment of copper minerals. L. F. CLARK, Assr. to UNION CARBIDE SALES CO. (U.S.P. 1,670,021, 15.5.28. Appl., 29.10.23).—Ores containing copper minerals are treated, prior to flotation, with acetylene and other oxidisable substances the oxidation of which results in the formation of insoluble substances. The particles of copper mineral are thus coated with a mixture of cuprous acetylide and the insoluble oxidation products which assist the subsequent flotation process.

A. R. POWELL.

Recovery of lead and zinc from zinc waste. E. BURY and F. W. WALKER (B.P. 290,035, 3.5.27).—A mixture of 10 pts. of zinc waste, 2 pts. of coke dust containing 2% S, 3 pts. of lime, and 2 pts. of coal is heated in a cement kiln by means of a flame of pulverised fuel burnt in an excess of oxygen, whereby the zinc is volatilised as oxide and the lead as sulphate. The residue in the kiln is suitable after grinding for the manufacture of hydraulic cement.

A. R. POWELL.

Metal [tungsten and molybdenum] alloys. BRIT. THOMSON-HOUSTON CO., LTD., Asses. of W. P. SYKES (B.P. 279,490, 21.10.27. U.S., 22.10.26).—Age-hardening alloys of iron with tungsten or molybdenum or both are prepared by heating together intimate mixtures of the constituent metals in a powdered form, such as is produced by the reduction of the oxides in hydrogen. The mixture is compressed into bars which are sintered in hydrogen above 1440° until diffusion is complete; the bars are then quenched and aged for several days at a temperature somewhat above 600°. The solid solubility of tungsten in iron at 1525° is about 33% and at 600° about 8%; that of molybdenum in iron is 24% at 1440° and 7% at 600°. The hardness of the quenched alloys is about 200–250 and that of the aged alloys, after annealing under the correct conditions, varies from 500 to 650 according to the composition.

A. R. POWELL.

Treatment of ores containing platinum. S. C. SMITH (B.P. 289,220, 10.3., 26.4., and 21.5.27).—Nickel-copper sulphide concentrates derived from South African norite deposits are roasted "sweet" and the product is heated with 70% sulphuric acid at 212° to dissolve substantially the whole of the copper and iron and the greater part of the nickel. The residue from this treatment is thoroughly washed and heated with 25–30% hydrochloric acid and a chlorate or manganese dioxide, whereby the platinum, palladium, gold, and part of the iridium and rhodium are dissolved and may be recovered by precipitation with zinc. Platinum is extracted from dunite ores by heating them at 500° and leaching with hydrochloric acid and an oxidising agent other than nitric acid.

A. R. POWELL.

Powder for welding castings, particularly of grey cast iron. F. POLITZ (U.S.P. 1,669,840, 15.5.28. Appl., 4.9.26. Ger., 21.10.25).—A small percentage of iron suboxide is added to the welding powder, as a flux.

F. G. CLARKE.

Soldering of metals or metal alloys. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 289,553, 31.1.27).—Tungsten or molybdenum may be soldered to other metals or alloys by means of a solder consisting of an alloy of copper with 2–3% Si or Al.

Borax or glass may with advantage be used as flux in the process.

A. R. POWELL.

Plant for extracting gold from sea-water. B. CERNÍK and B. STOCES (B.P. 289,638, 3.5.27).—A screen or lattice supported at some distance above the bottom of a natural or artificial reservoir for sea-water is covered with material capable of combining with or retaining gold, *e.g.*, pyrites or active charcoal, and channels are provided below the lattice to regulate the passage of the water through the material. A modified form for use in the open sea consists of an inverted U-tube containing a pump in one limb and the material in the other.

L. A. COLES.

Radioactive material. A. FISCHER (B.P. 278,347, 21.9.27. Austr., 29.9.26).—Oxides or other compounds of radioactive elements are mixed with an organic reducing agent, *e.g.*, tartaric acid or sugar, and an organic binder, and the mixture is applied to thin sheets or wires of a metal or alloy such as platinum or irido-platinum. The whole is then heated to such a temperature that the organic material is burnt off, and the radioactive coating is then incorporated in the surface of the carrying metal by mechanical treatment, *e.g.*, rolling, drawing, or hammering. Alternatively, the metal base may be used as a cathode in a fused electrolytic bath containing a compound of the radioactive element. For coating iron, steel, or silver sheet or wire the radioactive material may be incorporated with one of the usual mixtures used in producing a coating of fused enamel on the metal. The radioactivity of the resulting products is such that not only the γ -rays, but also the α - and β -rays, are fully utilised, and the products are capable of sterilisation by any of the usual methods.

A. R. POWELL.

Purification of metals. KAISER-WILHELM-INST. F. EISENFORSCHUNG (B.P. 265,213, 27.1.27. Ger., 27.1.26).—The metal to be purified is melted in a high-frequency induction furnace under a slag adapted to remove the impurities. Thus, nickel is refined under a slag containing lime, fluorspar, and nickel oxide, which removes sulphur, carbon, manganese, silicon, and aluminium, and is then deoxidised under a lime-fluorspar slag by the addition of the requisite quantity of silicon or manganese. Iron and iron alloys may be refined under a layer of hammer-scale and suitable fluxes. The rapid circulation of the metal caused by the high-frequency currents ensures a rapid and complete removal of impurities.

A. R. POWELL.

Production of aluminium by fusion electrolysis. I. G. FARBENIND. A.-G. (B.P. 283,949, 19.12.27. Ger., 21.1.27).—Alumina for addition to the electrolytic bath is rendered denser by grinding it under pressure as described in B.P. 272,109 (B., 1927, 591).

A. R. POWELL.

Case hardening [of iron]. A. LENNARTZ (U.S.P. 1,671,034, 22.5.28. Appl., 25.1.27. Ger., 15.2.26).—See G.P. 444,126; B., 1928, 57.

Production of [multi-coloured] gold strip or sheet. A. WHITEHOUSE (B.P. 289,660, 7.6.27).

Coating of tiles, sheets, etc. of iron or ferrous metals. A. REY (B.P. 270,298, 26.4.27. Fr., 27.4.26).

XI.—ELECTROTECHNICS.

Maintaining or restoring the electric capacity of the negative plates of lead accumulators. L. GAGNAUX (Scaled Note 1910, 30.6.09. Bull. Soc. Ind. Mulhouse, 1928, 94, 192—194). Report by E. BANDERET (*Ibid.*, 194—195).—The original capacity of the plates is maintained or may be restored (after reduction due to changes in the spongy condition of the lead forming the plate) by the addition to the electrolyte of the products obtained by the action of concentrated sulphuric acid on organic substances such as oils, fats, aromatic hydrocarbons, phenols, and resins; about 2—4 g. of the sulphonated product is required per litre of electrolyte. Alternatively, the organic substances may be incorporated in the negative plate before use so that they may be sulphonated during charging. Banderet reports favourably on the process.

A. J. HALL.

Cements and plastic masses. NAGEL and GRÜSS.—See IX. **Corrosion of metals.** HUDSON.—See X. **Electrolytic apparatus for analysis of insecticides etc.** HOLLAND and GILLIGAN.—See XVI. **Electrocolorimeter.** STOCKHAUSEN and WINDISCH.—See XVIII.

PATENTS.

[Electric] induction furnace. J. M. WEED, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,669,109, 8.5.28. Appl., 19.4.23. Renewed 18.8.27).—A primary winding and a secondary formed of a liquid charge contained in a looped cavity in a refractory receptacle are structurally related so that the spacing from the primary of sections of the secondary in planes passing through the axis of the primary increases progressively from one end to the other. A passage is provided external to the secondary through which a charge may circulate between portions of the secondary spaced at different distances from the primary.

J. S. G. THOMAS.

Production of electrical heating elements. E. L. and A. P. WIEGAND, Assrs. to E. L. WIEGAND Co. (U.S.P. 1,669,385, 8.5.28. Appl., 4.6.27).—A resistor is supported within, by, and between opposed parts of a sheath containing refractory electrical insulating material, and the sheath is reshaped so as to cause the insulating material to be distributed about the resistor after these opposed parts have been withdrawn therefrom.

J. S. G. THOMAS.

Magnetic materials. WESTERN ELECTRIC Co., Inc., Asses. of J. W. ANDREWS, R. GILLIS, A. F. BANDUR, C. P. BEATH, H. M. E. HEINECKE, and J. C. KARCHER (U.S.P. 1,669,642—9 and 1,669,665, 15.5.28. Appl., [A, B, E, F] 17.4.26, [C] 24.4.26, [D] 3.1.27. Renewed 16.2.28, [G] 3.1.27, [H] 10.4.26, and [I] 17.8.26).—(A—G) A series of compositions containing various granular magnetic materials (e.g., nickel-iron alloys) which are moulded and bound together by coatings of different insulating materials applied in successive layers (e.g., silicic acid and water-glass or ferric oxide, chromic acid and an alkali, a nitrate, kaolin and a flux, etc.). (H—I) A heat-treatment for rendering the above alloys brittle etc. is claimed.

J. S. G. THOMAS.

Magnetic core. G. W. ELMEN, Assr. to WESTERN ELECTRIC Co., Inc. (U.S.P. 1,669,658, 15.5.28. Appl.,

11.9.26).—A compressed mass of insulated particles of magnetic material having an initial permeability greater than that of iron is heat-treated to improve its magnetic properties.

J. S. G. THOMAS.

Storage-battery plate. J. H. CALBECK and J. A. SCHAEFFER, Assrs. to EAGLE-PICHER LEAD Co. (U.S.P. 1,668,801, 8.5.28. Appl., 26.7.24).—The formed paste of a storage-battery plate of high capacity, long life, and high porosity contains substantial amounts of fumed basic lead sulphate and of its derivatives formed in the development of the plate.

J. S. G. THOMAS.

Dry cell. E. H. McCABE, Assr. to FRENCH BATTERY Co. (U.S.P. 1,670,040, 15.5.27. Appl., 22.12.24).—A metal film is applied to the end part of a carbon electrode by means of an air blast directed upon the molten point of a metal wire, so that particles of metal freed from the wire are embedded in the pores of the carbon.

J. S. G. THOMAS.

Manufacture of carbons for electrotechnical uses. M. OSWALD (B.P. 289,680, 27.7.27).—Rough rupture surfaces bounded by smooth surfaces on carbon rods are provided with an adherent metallic or non-metallic covering.

J. S. G. THOMAS.

Battery electrolyte compound. Manufacture of storage batteries. L. R. STEELE (U.S.P. 1,668,740—1, 8.5.28. Appl., [A] 2.3.22, [B] 1.5.23).—(A) A suspension in water of a powdered compound formed from a soluble silicate and ammonium sulphate is used in place of sulphuric acid in storage batteries of the Planté type. (B) Sulphuric acid electrolyte is removed from a Planté-type cell after discharge to a voltage of about 1.7 and the electrolyte described under (A) is substituted.

J. S. G. THOMAS.

Manufacture of electrodes for arc welding. BRIT. THOMSON-HOUSTON Co., Ltd., Asses. of E. W. SCHWARTZ and F. R. KAIMER (B.P. 283,582, 4.1.28. U.S., 15.1.27).—Steel welding wire is boiled for 5—8 hrs. in a bath consisting of, e.g., water (100 pts.), sodium silicate (5 pts.), red oxide of iron (30 pts.), and lime (40 pts.) by wt.

J. S. G. THOMAS.

Manufacture of an electron-emitting cathode. E. E. SCHUMACHER, Assr. to WESTERN ELECTRIC Co., Inc. (U.S.P. 1,668,734, 8.5.28. Appl., 12.7.23).—Current is passed through a metallic filament supported in an agitated evacuated vessel containing powdered thermionically-active material.

J. S. G. THOMAS.

Manufacture of luminous electric discharge tubes. J. B. J. M. ABADIE (B.P. 276,018, 13.8.27. Fr., 14.8.26).—A discharge tube of glass containing salts of uranium or other fluorescent salts is filled with mercury and neon or helium.

J. S. G. THOMAS.

Devices for generating ultra-violet rays and activating oxygen etc. H. GEFFCKEN and H. RICHTER (B.P. 265,636, 8.2.27. Ger., 8.2.26).—Ultra-violet radiation is produced by a glow discharge between mercury electrodes in a discharge vessel of material, e.g., quartz, transparent to the radiation.

J. S. G. THOMAS.

Absorption of non-precious gases in manufacturing electric discharge tubes. E. SCHRACK and R. ETENREICH (RADIOWERK E. SCHRACK) (B.P. 272,843, 18.11.26. Austr., 17.6.26).—Non-precious gases, e.g.,

oxygen, nitrogen, are removed from the tubes by heating therein to about 150° an alloy of rare-earth metals and a relatively large amount of heavy metals, e.g., an alloy containing 50% Ce, 25% Fe, 20% La, and 5% Di.
J. S. G. THOMAS.

[Electric] induction furnace having unidirectional circulation. J. R. WYATT, Assr. to AJAX METAL CO. (Re-issue 16,967, 15.5.28, of U.S.P. 1,312,069, 5.8.19).—See B., 1919, 687 A.

Electrode mass for self-baking electrodes. C. W. SÖDERBERG, Assr. to NORSKE A./S. FOR ELEKTROKEMISK IND. (U.S.P. 1,670,052, 15.5.28. Appl., 22.7.24. Norw., 25.7.23).—See Swiss P. 111,653; B., 1926, 284.

Manufacture of battery plates. W. J. PLEWS, Assr. to PLEWS & HUTCHINSON CORP. (U.S.P. 1,670,046—7, 15.8.28. Appl., [A] 27.6.21, [B] 29.12.27).—See B.P. 207,606; B., 1924, 101.

Aluminium (B.P. 283,949). Radioactive material (B.P. 278,347).—See X. **Electrodeposition of rubber (B.P. 289,965).**—See XIV.

XII.—FATS; OILS; WAXES.

Determination of the degumming powers of soaps [for boiling-off silk]. R. TSUNOKAE (J. Soc. Dyers and Col., 1928, 44, 142—144).—The degumming action of a soap on raw silk being proportional to its degree of hydrolysis in aqueous solution, the degrees of hydrolysis shown by a number of commercial soaps as determined by the salting-out, titration, toluene, and electro-conductivity methods were compared and correlated with their degumming powers. The salting-out method proved unsatisfactory, but the other methods yielded comparable results, except that the toluene method, in which the free fatty acid liberated by hydrolysis is extracted with toluene and afterwards weighed, yielded somewhat low results. The toluene method yields unreliable values for hydrolysis when applied to soaps containing oxidised fatty acids. The degumming powers and degrees of hydrolysis of sodium stearate, palmitate, and oleate decrease in the order named.

A. J. HALL.

Oils of the chaulmoogra group. Gorli fat. E. ANDRÉ and D. JOUATTE (Bull. Soc. chim., 1928, [iv], 43, 347—360).—The fat obtained in 39—53% yield from seeds of the *Oncoba echinata* (gorli) plant after 18—20 recrystallisations from acetone and light petroleum affords a solid glyceride, m.p. 51°, $[\alpha]_D +49.33^\circ$, saponif. value 194°, iodine value 72.5, together with a mixture of liquid glycerides, d_{40}^{16} 0.9482, n_D^{20} 1.4828, $[\alpha] +46.4^\circ$, saponif. value 194, iodine value, 108. Comparison of the properties of the solid glyceride with those of synthetic *trichaulmoogrin*, m.p. 45°, n_D^{20} 1.4764, d_{40}^{16} 0.9437, $[\alpha] +54.83^\circ$, saponif. value 190.6, iodine value 87.3, indicates that the solid glyceride is probably a molecular compound of *trichaulmoogrin* and *palmitodichaulmoogrin*, palmitic acid being present to the extent of about 15%. The crude fat contains 10—12% of palmitic acid, about 80% of chaulmoogric acid, and about 10—12% of a liquid unsaturated acid which appears to contain two ethylenic linkings, and for which the name *gorlic acid* is proposed. The new acid, which

has not been obtained perfectly pure, has d_{40}^{18} 0.9364, n_D^{20} 1.4783, $[\alpha] +50.3^\circ$, iodine value 169.6 (Cheery 181.4) (cf. Dean and Wrenshall, B., 1921, 89 A; 1925, 928). The *amide*, m.p. 95°, *chloride*, *diethylamide*, *lithium salt*, and *gorlicohydroxamic acid* have been prepared. *Gorlic acid* contains the pentamethylene ring characteristic of the acids of the chaulmoogric group, and it is probable that one ethylenic linking is present in the ring system and one in the side chain. *Trichaulmoogrin* could not be isolated by extraction of *gorli fat*, and its m.p. constitutes an exception to the rule that a triglyceride melts at a higher temperature than the constituent fatty acid.
R. BRIGHTMAN.

Rate of dissolution of essential principles during percolation. II. Extraction of raffinose from cotton-seed cake. M. BRIDEL and (Mlle.) M. DESMAREST (J. Pharm. Chim., 1928, [viii], 7, 433—447, and Bull. Soc. Chim. biol., 1928, 10, 510—521; cf. B., 1928, 388).—Raffinose (300 g.) is best obtained from cotton-seed cake (10 kg.) by threefold percolation with 60% alcohol (3000 c.c. each time) at the ordinary temperature followed by removal of the alcohol, dilution with water, and defecation with lead salts. The extraction removes 92.93% of the total raffinose (7.2%) in the first 5 hrs., and is complete in 10 hrs. Inversion of the sugars in the final liquors shows that other optically active substances are present (cf. Hudson and Harding, A., 1914, i, 1166).
G. A. C. GOUGH.

Flour from linseed from which the oil has been expressed. E. ANDRÉ (J. Pharm. Chim., 1928, [viii], 7, 481—486).—Possible methods of utilising to the best advantage linseed grain are discussed, special reference being made to the use in pharmacy of the flour obtained after expressing the oil and separating the husk.
W. O. KERMACK.

Auer's conception of the drying of oils. A. EIBNER (Farben-Ztg., 1928, 33, 2165—2169).—The "gas-coagulation" theory is criticised mainly on the grounds that in its evolution Auer has ignored the work of earlier investigators on the individual characteristics of various oils, the hydrophilic nature of linseed oil films, etc. Some of the author's experiments in this field are briefly recapitulated.
S. S. WOOLF.

Standardisation of methods of testing oil. Measuring flasks for Engler's viscosimeter. H. SCHLÜTER (Chem.-Ztg., 1928, 52, 367).—For the standardisation of the Engler viscosimeter with water the collecting flask is usually graduated to deliver a certain volume so that several tests can be done successively. This graduation is useless when testing oils, as the amount which remains in the flask on draining varies with the oil. Hence it is recommended that these flasks should be graduated for collecting and for delivering a standard volume.
A. R. POWELL.

Stearic acid standards. ANON. (Soap, 1927, 3, 37).

Ethylene dichloride as a solvent. BAUER and LAUTH.—See III.

PATENTS.

Degreasing of bones and other materials containing moisture. BRIT. GLUES & CHEMICALS, LTD.,

and R. B. DREW (B.P. 289,569, 3.2.27).—By means of the apparatus described it is possible to degrease bones with hot vapour of a volatile solvent, at the same time vaporising the water content of the bone. By an arrangement of several extracting vessels the mixed solvent and water vapours are used for further extractions until its solvent properties are exhausted by the increasing proportion of water vapour, after which the solution of fat is separated from the water and the solvent evaporated for further use. E. HOLMES.

Treatment of fats and oils. H. T. BÖHME A.-G. (B.P. 281,232, 7.10.27. Ger., 25.11.26).—Sulphuric acid which is extensively used in preparing water-soluble fatty sulphonates or alkali sulphonates may be replaced by phosphoric acid in preparing compounds of value in the textile, leather, and pharmaceutical industries. Thus, the oil or fat is treated with phosphoric anhydride, washed to remove the excess phosphoric acid, and then preferably converted into the alkali salt. The reaction may be assisted by the addition of acetic anhydride or acetyl chloride, or by the employment of acetylphosphoric acid. It is claimed that the process gives a product of much better colour than the sulphuric acid process. E. HOLMES.

Manufacture of soap. A. E. HATFIELD, E. A. ALLIOTT, and ACHILLE SERRE, LTD. (B.P. 289,582, 15.2.27).—Soaps of especial value in dry-cleaning work may be prepared by wholly or partially saponifying a fatty acid of setting point below 2°, preferably below -0.5°, with aqueous ammonia or solutions of potassium or sodium carbonates or hydroxides, an emulsifying agent being added at some stage of the manufacture. Various emulsifying agents, as di- and tri-chloroethylene and pentachloroethane, may be used, whilst if cyclohexanol be employed the setting point of the original acid may be as high as 10°. Practically pure oleic acid is suggested as a suitable starting material. E. HOLMES.

Production of preparations capable of being emulsified and forming soaps. G. PETROFF (B.P. 289,934, 1.2.27).—Sulpho-aromatic fatty acids of high mol. wt. are obtained by treating mixtures of liquid aromatic hydrocarbons of low mol. wt. and saturated fatty acids or stearine with sulphuric acid containing free sulphur trioxide. The products emulsify in the presence of dilute mineral acids, and give light-coloured soaps. [Stat. ref.] B. FULLMAN.

Production of cleansing and degreasing agents and emulsifying agents. R. SAJITZ and E. POTT (CHEM. FABR. POTT & Co.) (B.P. 266,746, 25.2.27. Ger., 25.2.26).—Aromatic alcohols which are difficultly soluble or insoluble in water, and in particular benzyl alcohol, are treated alone or in combination with hydrocarbons (e.g., solvent naphtha) with alkali salts of fatty acids, sulfo-fatty acids, naphthenic acids, sulphonates of aromatic or of hydroaromatic hydrocarbons, or corresponding acids. Benzyl alcohol does not affect the frothing capacity of such agents, and has the advantage of possessing only a slight odour. E. HOLMES.

Generation of foam from foam-developing substances. R. SCHNABEL, ASSR. to MINIMAX A.-G. (U.S.P. 1,669,213, 8.5.28. Appl., 23.4.27. Ger.,

30.9.25).—A saponin solution is supersaturated under pressure with a gas which is readily absorbed and which, on removal of the pressure, is liberated again with the development of foam from the liquid. A. R. POWELL.

Melting [blocks of] fatty substances. SOC. ANON. ANC. ÉTABL. G. PELLERIN (B.P. 269,873, 24.3.27. Fr., 22.4.26).

Manufacture of soap flakes. MASCHINENFABR. ELITE A.-G. (B.P. 268,320, 9.3.27. Ger., 29.3.26).

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

The red lead question. L. SCHERTEL (Farben-Ztg., 1928, 33, 2101—2104).—Mainly polemical with Rahder (cf. B., 1928, 99). Accelerated tests indicate that "disperse" red lead is in no way inferior to the "classic" red lead as a protective pigment. S. S. WOOLF.

Production of flat oil varnishes. F. WILBORN (Farben-Ztg., 1928, 33, 2169—2170).—Various substances are added to oil varnishes to produce a "matt" finish, e.g., waxes, aluminium hydroxide, magnesium carbonate, kaolin, aluminium stearate, palmitate, resinate, zinc stearate, etc. The properties and suitability of the various types are discussed. S. S. WOOLF.

"Cellosolve" [ethylene glycol monoethyl ether] and its derivatives in nitrocellulose lacquers. E. W. REID and H. E. HOFMANN (Ind. Eng. Chem., 1928, 20, 497—504).—The properties of "cellosolve," "cellosolve acetate," and ethylene glycol monobutyl ether are described, and the advantages these compounds possess over other nitrocellulose solvents in the manufacture of nitrocellulose lacquers are discussed. Resins and combinations of resins suitable for use with these solvents are indicated, also the factors to be considered in the selection of solvent mixtures and thinners. Typical working formulæ are given for nitrocellulose lacquers for special purposes. D. J. NORMAN.

Plastic masses in electrotechnics. NAGEL and GRÜSS.—See IX.

PATENTS.

Manufacture of non-thickening paints and lacquers. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 289,968, 7.2.27. Addn. to B.P. 289,153; B., 1928, 456).—The process of the prior patent is extended to paints and lacquers having a pigment base of zinc compounds other than lithopone. L. A. COLES.

Manufacture of lacquers, impregnating materials, etc. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 289,523, 26.1.27. Addn. to B.P. 286,789; B., 1928, 376).—Cellulose esters and ethers, especially in the manufacture of films etc., are dissolved in compounds of the acetal type of polyhydric alcohols in the absence of water. B. FULLMAN.

Manufacture of lithopone. W. H. LANDERS (U.S.P. 1,669,476, 15.5.28. Appl., 21.2.25).—Undried lithopone is fed into the higher end of a slightly inclined rotating tubular furnace in which it passes successively through drying, preheating, and calcining zones. Combustible gas is introduced into the furnace at the lower end and is burnt in the presence of insufficient air for complete

combustion, whilst more air is introduced between the calcining zone and the higher end of the furnace.

L. A. COLES.

Manufacture of crude lithopone. J. H. CALBECK, Assr. to EAGLE-PICHER LEAD Co. (U.S.P. 1,669,857, 15.5.28. Appl., 21.1.26).—Aqueous solutions of zinc sulphate and barium sulphide are charged continuously in equivalent proportions into a mixing tank, and the overflow passes into a settling tank. L. A. COLES.

Preparation of phthalic anhydride-glycerol resins. J. H. SCHMIDT, Assr. to BAKELITE CORP. (U.S.P. 1,663,183, 20.3.28. Appl., 28.4.24).—The colourless product obtained by the action of phthalic anhydride on a polyhydric alcohol below 175° is heated, at temperatures below the formation of inherent colour, until it is hard, tough, and infusible. B. FULLMAN.

Manufacture of a phenol[-formaldehyde] resin. C. J. ROMIEUX, Assr. to AMER. CYANAMID Co. (U.S.P. 1,669,674, 15.5.28. Appl., 29.4.25).—A reactive resin is prepared by reaction between a phenol of b.p. above 202° and formaldehyde, in the presence of alkali (less than 0.1% based on the phenol present).

B. FULLMAN.

Production of artificial resin. C. KULAS (U.S.P. 1,669,831, 15.5.28. Appl., 16.10.25).—Phenol-aldehyde resins, after separation from the reaction mixture by cooling and settling, are centrifuged in the cold condition. The layers of partially dehydrated resins which subsequently separate are subjected to rotation while supported upon a heated surface. The resin layers finally separating are centrifuged cold.

F. G. CLARKE.

Production of solid, finely-divided material [litharge]. L. SCHERTEL and W. LÜTY, Assrs. to T. GOLDSCHMIDT A.-G. (U.S.P. 1,670,049, 15.5.28. Appl., 14.11.22. Ger., 15.11.21).—See B.P. 189,132; B., 1924, 343.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Effect of heat on raw rubber. C. R. PARK, C. M. CARSON, and L. B. SEBRELL (Ind. Eng. Chem., 1928, 20, 478—483).—Heating with water in an autoclave for 24 hrs. at 158° plastifies rubber to approximately the same extent as passage five times between tight rolls. The proportion of the acetone-soluble matter becomes increased, as also do its acid number and its nitrogen content; these changes are attributable to hydrolysis of protein and fatty constituents and to depolymerisation of rubber itself. The rate of vulcanisation of the rubber is increased for unaccelerated mixings and for mixings containing mercaptobenzthiazole, but is not appreciably altered for hexamine, diphenylguanidine, and ethyldeneaniline; the stress-strain curves of the vulcanised products, relative to similar products from unheated rubber, are more convex to the strain axis. The heat-treatment has no adverse effect on the ageing qualities and sometimes improves them. It is probable that amines, nitrogen bases, and alkaloids are the substances mainly responsible for the characteristics of heated rubber. D. F. TWISS.

"Normal ageing" of compounded rubber. R. H. MCKEE and H. A. DEFEW (Ind. Eng. Chem., 1928, 20,

484—491).—Nine vulcanised mixtures of different composition have been examined after ageing under various conditions. Storage in a wet atmosphere gives a softer stress-strain curve than storage in a dried atmosphere; drying after a long exposure to a moist atmosphere causes an increase in tensile strength. Over-vulcanised rubber deteriorates in tensile strength and also oxidises more rapidly in a dry than in a wet atmosphere; under-vulcanised rubber decreases in tensile strength less rapidly in dry air than in moist, but the rate of oxidation is approximately the same in both. Paraffin wax exerts a protective action against sunlight with samples under strain; zinc oxide also is a protective agent against sun-cracking, doubtless because of its opacity. Storage of mixed rubber for 1½ or 3 years causes marked reduction in rate of vulcanisation and slight decrease in tensile strength. D. F. TWISS.

Influence of fatty acids on vulcanisation [of rubber]. G. S. WHITBY and B. A. EVANS (J.S.C.I., 1928, 47, 122—126 r).—Accelerators requiring zinc oxide for activation need this in a soluble form; the presence of organic acids is not immaterial in the presence of a plentiful supply of zinc oxide (cf. Martin and Davey, B., 1925, 602). Mercaptobenzthiazole produced no vulcanisation at 126° in the absence of the resin acids even with deresinated rubber and zinc oxide in equal proportions, whereas with the addition of oleic acid (1½ pts. : 100 pts. of rubber) good vulcanisation was obtained in 30 or 40 min. Stearic acid also exercised as favourable an effect on the activity of anhydroacetaldehyde-aniline in the presence of 20 pts. as of 5 pts. of zinc oxide. In the case of accelerators, such as hexamethylenetetramine and diphenylguanidine, which are capable of inducing good vulcanisation in the absence of zinc oxide, the presence of organic acids is relatively unimportant. Of the three fatty acids naturally present in rubber, the effectiveness in assisting vulcanisation decreases in the order: stearic, oleic, linoleic; the lower effectiveness of the unsaturated acids may be attributable to their combination with sulphur and consequent reduction of its active mass. D. F. TWISS.

Rubber vulcanisation accelerators in ultra-violet light. R. DITMAR and W. DIETSCH (Chem.-Ztg., 1928, 52, 388—389).—Some organic accelerators give characteristic fluorescence when exposed to the light from a quartz-mercury lamp after passage through a "uvioi" filter: fluorescence is also exhibited by raw or vulcanised mixings containing certain accelerators. As an analytical method, however, for purposes of identification, the method would be of value only in conjunction with other tests. D. F. TWISS.

Colouring of cold-cured rubber. W. E. SANDERSON (J. Soc. Dyers and Col., 1928, 44, 137—140).—The use of organic accelerators (e.g., tetraethylthiourea disulphide, zinc diethyldithiocarbamate, and zinc isopropyl-xanthate) in the vulcanisation of rubber allows brighter shades to be obtained from dyes previously used for colouring rubber, and has extended the range of dyes suitable for this purpose. Cold-cured rubber goods may be coloured by immersion in aqueous solutions of basic dyes at 88° and drying by drumming in warm air with the addition of sawdust for removal of the loose

colour, or by dipping in a solution of oil-soluble dyes in benzene or solvent naphtha containing a small amount of stearic acid, and then evaporating the solvent. Many basic, direct, sulphur, and vat dyes are readily absorbed by rubber latex from their aqueous solutions on the addition of acetic acid, it being desirable to add tannic acid before the acetic acid when basic dyes are used; the colour of the resulting coagulated latex resists subsequent cold-cure processes, the acid dyes being generally most resistant. Dyes or colour lakes used for rubber goods vulcanised by the cold-cure process should be fast to acids, since free acid is formed during this process by the action of moisture on the sulphur monochloride used. Lake pigments are generally suitable for "spread work" such as rubber-coated fabrics, and comprise acid dyes precipitated by barium chloride; basic dyes precipitated by tannic acid, Katanol (a thiophenol), Tamol (a condensation product of naphthalenesulphonic acid and formaldehyde), or phosphotungstic or phosphomolybdic acid (these lakes have superior fastness to light); the insoluble azoic pigments; alizarin dyes precipitated on alumina; and insoluble earth colours. Aluminium bronzes are used in producing metallic effects.

A. J. HALL.

Thermal and calorimetric magnitudes of caoutchouc and of similar substances. M. LE BLANC and M. KRÖGER (Z. Elektrochem., 1928, 34, 241—244).—The specific heats of raw and vulcanised rubber in both the normal and stretched conditions have been determined at temperatures from about -170° to 40° . The significance of the irregular temperature-specific heat curves is discussed.

H. F. GILLBE.

PATENTS.

Preparation of latex products for rubber manufacture. J. E. NYROP (B.P. 289,583, 15.2.27; cf. B.P. 259,922; B., 1928, 41).—Using the spraying apparatus described earlier, latex is completely or partly dried, anti-coagulating agents such as potash soap and also, if desired, softening agents, *e.g.*, oils, and other compounding ingredients, being simultaneously sprayed without previous mixing with the latex. The products are capable of being redispersed or softened with water and are suited to further treatment in rubber manufacture.

D. F. TWISS.

Electrodeposition of rubber and the like from latex. W. A. WILLIAMS (B.P. 289,965, 7.2.27).—Into alkaline latex containing an added electrolyte such as ammonium chloride are introduced two suitable electrodes which are conductively connected outside the liquid; the resulting galvanic action causes electro-negative rubber globules to deposit on the anode; *e.g.*, commercial preserved latex, approximately 0.5*N* with respect to ammonia, is mixed with twice its volume of a saturated solution of ammonium chloride, and the electrodes consist, respectively, of a zinc plate and a carbon element surrounded with manganese dioxide. Compounding ingredients may be added to the latex so as to be deposited with the rubber.

D. F. TWISS.

Dispersion of rubber into a colloidal substance. H. L. TRUMBULL and J. B. DICKSON, Assrs. to B. F. GOODRICH Co. (U.S.P. 1,668,879, 8.5.28. Appl., 23.4.26).

—Rubber, containing a tacky softener, is milled into a viscous paste of a substance such as glue, casein, or gluten, so as to produce a dispersion of this in rubber, the dispersion being subsequently inverted so as to bring the paste into the continuous phase.

D. F. TWISS.

Vulcanisation of rubber to leather. L. B. CONANT (B.P. 275,194, 5.7.27. Can., 29.7.26).—Leather after being preheated in air to approximately the vulcanisation temperature is assembled, while still hot, with the rubber composition, which is then vulcanised under pressure by heat.

D. F. TWISS.

Rubber articles. L. C. PETERSON (B.P. 283,122, 3.1.28. U.S., 3.1.27).—Rubber sheets, with one side vulcanised and the other side raw, suitable, *e.g.*, for tyre patches, are prepared by superposing a layer of a mixture of rubber including a vulcanising agent and an accelerator on a layer of rubber mixture free from vulcanising ingredients and containing an acidic substance such as sodium hydrogen sulphate. When the composite sheet is heated to vulcanise the backing the sodium hydrogen sulphate prevents the raw facing from becoming vulcanised by any sulphur which may migrate from the backing.

D. F. TWISS.

Manufacture of reinforced abrasive articles [containing rubber]. W. B. WESCOTT, Assr. to RUBBER LATEX RES. CORP. (U.S.P. 1,668,475—6, 1.5.28. Appl., [A] 18.11.22, [B] 16.9.25. Renewed 11.2.28).—(A) A mixture of comminuted old rubber with abrasive materials, sulphur, and latex is dried, moulded, and vulcanised. (B) An abrasive article is formed of granular abrasive material held together by a cementing layer which also binds irregularly disposed reinforcing fibres; the cementing layer also comprises a continuous bond of rubber.

D. F. TWISS.

Production of rubber compositions. H. L. FISHER, Assr. to B. F. GOODRICH Co. (U.S.P. 1,668,235—7, 1.5.28. Appl., [A] 14.1.24. Renewed 14.3.28, [B] 17.1.24, [C] 14.4.24. Renewed 14.3.28).—(A) Raw rubber is treated with *p*-toluenesulphonic acid. (B) Concentrated sulphuric acid (2—25 pts.) mixed with an inert carrier is milled into rubber (100 pts.) and the mixture is heated to reaction temperature. (C) A mixture of rubber (100 pts.) with a sulphonic acid or chloride (8 pts.), *e.g.*, *p*-toluenesulphonic acid, and with sulphuric acid (2 pts.) is heated so as to cause a pronounced exothermic reaction.

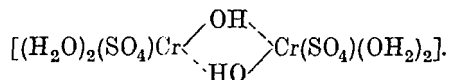
D. F. TWISS.

Rubber composition. F. C. DYCHE-TEAGUE (U.S.P. 1,670,599, 22.5.28. Appl., 19.2.26. U.K., 10.12.25).—See B.P. 268,853; B., 1927, 452.

XV.—LEATHER; GLUE.

Chrome tanning. VI. Properties and behaviour of various pretreated chromium sulphate solutions. E. STIASNY and O. GRIMM (Collegium, 1928, 49—71; cf. B., 1927, 393).—The p_H value of a 1% solution of hexa-aquochromium sulphate, 0% basicity, which had not been heated was 2.81 and gradually diminished to 1.98 on being kept for 4 months. The "verolung," which increases the p_H value, is outweighed by the effect of the migration of the sulphate residues

in the chromium complex. The addition of hydrochloric acid to the above solution restrained the sulphate residue migration, and the acidity of the liquors was less than the calculated value. When the chromium sulphate solution was boiled for 5 min. the p_H value fell to 1.21, but increased to 1.81 after being kept for 4 months. The diminution in p_H value was much greater than for chloride solutions and pointed to much greater "verolung" with sulphate solutions, but the increase in p_H value on keeping was also greater with chromium sulphate than with chromium chloride solutions. The "verolte" basic sulphate returns to its original state more easily than the chloride. There was evidence on adding hydrochloric acid to the boiled chromium sulphate solution that the (SO_4) ions exercise a neutral salt effect and slightly diminish the p_H value. Boiling the chromium sulphate solution for longer periods did not cause much further change; thus, the p_H value after 60 hrs. was 1.20. In this it differs from chromium chloride liquors. On adding hydrochloric acid to the boiled solution and keeping it, the p_H value increased due to the reversal of the "verolung" process. Dilution caused a diminution in the acidity of cold prepared solutions and a slight diminution in that of both boiled solutions which can be explained by "verolung." The acidity of a 33% basic chromium sulphate solution which had not been heated increased on being kept. This is explained by "verolung" of the monohydroxosulphate which disturbs the hydrolysis equilibrium and so forms more acid. The addition of hydrochloric acid to the basic chromium sulphate solution caused a diminution in the acidity which diminished still more on ageing, this again being due to reversal of the "verolung" process. Boiling the solution caused an increase in the acidity due to greatly increased hydrolysis, progressive "verolung," and diminished buffering action by the (SO_4) ion, but the acidity gradually fell to that obtained on ageing the cold solution. These and other researches show that "verolung" plays the greatest rôle in chromium sulphate solutions. There is no evidence of the formation of oxygen bridges, as with chromium chloride solutions, owing to the greater tendency of the (SO_4) residues to form complexes of the type.



The migration of the acid radical is only found in 0% basic chromium chloride solutions, but it is different with chromium sulphate solutions. The buffering action of the chlorine ion is not observed with basic salts, but the (SO_4) ion always exerts a buffering effect due to the formation of (SO_4H') ions. Frequently this effect exceeds all the other factors affecting acidity. Dialysis tests, hide powder tests, and determinations of the precipitation value throw little light on the properties of the liquors.

D. WOODROFFE.

Chrome tanning. VII. Hydrolysis and tanning action of sulphato-chromium sulphates. E. STIASNY and D. BALÁNYI (Collegium, 1928, 72—97).—It is shown that sulphato-chromium sulphates are less hydrolysed, and therefore less acid, than hexa-aquo-

chromium sulphates. "Verolte" basic sulphato-chromium sulphates are obtained on boiling hexa-aquo-chromium sulphate solutions. These solutions are more acid than those of the hexa-aquochromium sulphates owing to the increased acidity consequent on the "verolung." (SO_4) radicals always enter into the chromium complex. This migration is always hastened by adding alkali, *i.e.*, making the liquor basic, thus showing that "verolung" and masking of the (SO_4) groups are parallel processes. The amount of both masked and ionised (SO_4) groups combined with chromium diminishes with increasing degree of basicity. The relative amount of sulphate groups in the complex remains fairly constant meanwhile. If a solution of chromium sulphate is rendered basic with sodium carbonate instead of with sodium hydroxide, no dihydroxo-chromium sulphate is precipitated, and a much greater basicity is attained before precipitation commences. On adding sodium carbonate, carbonate radicals enter the chromium complex as well as hydroxo- and "ol" groups. These carbonate groups stimulate "verolung," and in consequence of the increased "verolung" there is much more migration of (SO_4) groups into the chromium complex. The increased number of such groups represses the carbonate groups, and finally, in a chrome liquor rendered highly basic with sodium carbonate, there are very few carbonate groups in the complex. When a solution of hexa-aquo-chromium sulphate was boiled, "verolung" took place and a portion of the sulphate radicals combined as ions migrated into the complex. This process was very rapid at b.p. owing to the disturbance of the hydrolysis equilibrium by "verolung." On cooling, there is a reversal of the "verolung" process due to the acid formed, and some of sulphate radicals in the complex become sulphate ions again. The acid present reacts with the latter forming (HSO_4) ions, and thus the acidity diminishes. This latter process is only subsidiary in feebly basic solutions, but if the boiled solution is immediately cooled rapidly and rendered basic, the free acid will be neutralised and the reversal of the "verolung" on keeping is hindered. This effect is obtained with liquors of 16.6% basicity, which are prepared by adding the necessary amount of sodium hydroxide which corresponds to the amount of acid liberated. Consequently, the p_H value of the solution increases only slightly on ageing. The small increase is due to the acid formed by the hydrolysis of the basic sulphato-sulphate, which acid has a slight tendency to reverse the "verolung." The proportion of sulphate radicals in the complex to those present as ions is not affected to the same extent by rendering the liquor basic. Large amounts of sulphate radicals entered the chromium complex as a result of boiling, but they gradually returned to the ionic form on being kept. Since the temperature coefficient of the formation of sulphate complexes is large, many sulphate groups gradually return to the ionic form, though a number of masked sulphate groups remain even after seven weeks. In a basic liquor the free acid formed by hydrolysis is neutralised, and there is therefore less reversal of the "verolung" process. The presence of hydroxo-groups in basic salts does not diminish the masked (SO_4) groups

([SO₄] in the chromium complex), and these are the same in both the basic and normal liquors. Tanning tests with different solutions of chromium sulphate show that the chromium complexes in the tanned hide powder contain more sulphate groups than those in the chrome liquor. This is attributable to the stronger tanning power of the chromium complexes containing more sulphate groups, and which are at the same time more strongly "verolten." Both types of hydroxy-groups, "verolten" and not "verolten," are necessary to tannage. D. WOODROFFE.

Determination of sulphato-groups in chrome-tanned leather. H. B. MERRILL, J. G. NIEDERCORN, and R. QUARCK (J. Amer. Leather Chem. Assoc., 1928, 23, 187—208).—The curve showing the rate of removal of sulphuric acid from chrome-tanned leather by pyridine solution indicates that some acid combined with the chromium is also removed, hence Gustavson's method (B., 1927, 261) of determining the acidity of chrome leather is wrong. Experiments with chrome leather and water at different p_H values respectively have shown that p_H 5.4 or slightly lower is the maximum acidity at which protein-bound acid can be removed completely, and the following method, based on this, is recommended. A sample (2 g.) of the chrome leather is weighed out into a shake bottle, covered with 100 c.c. of distilled water, shaken for 1 hr., and allowed to remain overnight. Next day, 2 drops of 0.5% methyl-red solution are added and the contents are titrated with 0.02*N*-sodium hydroxide until the colour changes from clear red to salmon, i.e., p_H 5.2—5.4. The mixture is then shaken for 15 min., restored to the proper shade for p_H 5.3 by adding alkali, and shaken for the rest of the day. The shade is examined every 15 min. for the first 2 hrs. and every 30 min. after that. After being kept overnight, it is neutralised to p_H 5.3 four times more at intervals of 1 hr. The entire neutralisation should occupy less than 48 hrs. The solution is poured off, and the leather transferred to a Wilson-Kern extractor, washed with running distilled water for 1 hr., again transferred to a beaker, and the sulphate remaining in the sample determined by the phosphate displacement method, thus obtaining the chromium-bound sulphate.

D. WOODROFFE.

Glue testing. Report of the Commission of the German Association for testing technical materials. O. GERNGROSS (Collegium, 1928, 130—143).—Ten samples of commercial glues were tested. The moisture was determined by heating 3—5 g. of the finely-divided material at 100—105° for 18 hrs., or to constant weight under atmospheric pressure. Phosphorus pentoxide should be used in all desiccators. The results showed that the best glue contained 17% of water. The toluene distillation method decomposes the glue. The p_H value of a 1% solution was determined colorimetrically, but did not give a constant potential in the electro-metric method. Good agreement was obtained in the viscosity determinations on a 17.75% solution at 40°, using the Engler viscosimeter. The viscosity was also determined by the Ostwald apparatus on 3% and 10% solutions. The report favours the Vogel-Ossag apparatus for gelatin and glue solutions. The jelly strength as determined by the American apparatus

showed fairly large individual variations, and is rejected as being too crude. The Greiner apparatus (A., 1927, 19) was used on 10% solutions which had been left 16 hrs. at 18—20°, and gave better concordance. The adhesive properties of the different glues, tested on pieces of wood, depended on the glue concentration, and variations were attributed not only to molecular and micellar phenomena, but also to the diffusibility of glue into the wood. A good, highly viscous, quick-setting glue will diffuse less into the wood than poor thin glue. The poorer glue will compare more favourably in concentrated solutions. Glues should be diluted to 30—40% strength and their adhesive properties tested by the Rudeloff process (cf. B., 1918, 743 A). The Bechhold-Neumann test (B., 1924, 759) is criticised. Some of the glues tested presented anomalies of bad viscosity and joint strength, with very good jelly strength. One such glue was derived from chrome-tanned leather shavings. Results with the different glues showed that the Greiner jelly-strength test (*loc. cit.*) showed better agreement with the joint-strength tests than did the viscosity determinations.

D. WOODROFFE.

Theory of leather dyeing. SALT.—See VI. **Gelatin for ice cream.** DAHLBERG and others.—See XIX.

PATENTS.

Preparation of fish skins for making "shagreen."

H. V. ANDREW, and G. BETJEMANN & SONS, LTD. (B.P. 289,213, 7.3.27).—Skins of ray fish and other fish are cleaned, dyed, or stained, brought to an even thickness, and a stencil pattern is applied to the back of the skin, which is then sprayed with pigment or colour. The front of the skin is finished or polished as usual.

D. WOODROFFE.

Treatment [bleaching] of fur skins and the like.

H. STEIN, W. E. AUSTIN, and I. LIEBOWITZ, ASSRS. to STEIN FUR DYEING Co., INC. (U.S.P. 1,668,875, 8.5.28. Appl. 14.10.25).—The skins are treated in a solution containing ammonia, a soap soluble in water, and more than 3% of hydrogen peroxide.

A. R. POWELL.

Treatment of fats and oils (B.P. 281,232).—See XII. **Rubber on leather** (B.P. 275,194).—See XIV.

XVI.—AGRICULTURE.

Soil experiments. O. LIEHR (Z. Pflanz. Düng., 1928, 7B, 201—206).—Nutrient values of a large number of soils were determined by the Neubauer seedling method and by that of Lemmermann and König. No general relationship existed between these values and the soil reaction. A general lack of phosphate in German soils is indicated.

A. G. POLLARD.

Relative lime needs of ammonium sulphate and sodium nitrate, and of different crops. B. L. HARTWELL and S. C. DAMON (J. Amer. Soc. Agron., 1927, 19, 843—849).—Each 100 g. of ammonium sulphate requires about 120 g. of limestone more than is required by 128 g. of sodium nitrate to attain equal soil reaction. In general, crop yields are similar for the two sources of nitrogen and the same reaction.

CHEMICAL ABSTRACTS.

Utilisation of nitrogen by grass land in the North Sea marshlands. F. TRILLING (Z. Pflanz. Düng.,

1928, 7B, 226—234).—Considerable variation in the efficiency of nitrogenous manures in marshes is recorded. Newer soils respond more markedly to treatment with ammonium sulphate than do older ones. Increases in hay yields following nitrogenous fertilisation are greater in soils having little clover in the herbage. Botanical examination shows this increase is largely in the proportion of meadow grass, with a diminution in that of dogstail. The relatively small effect of ammonium sulphate on rich clover land is ascribed to the influence of the different type of herbage on the physical and chemical properties of the soil. A. G. POLLARD.

Organic food reserves in relation to the growth of lucerne and other perennial herbaceous plants. L. F. GRABER, N. T. NELSON, W. A. LUEKEL, and W. B. ALBERT (Wisconsin Agric. Exp. Sta. Res. Bull., 1927, [80], 128 pp.).—The maturity and quantity of top growth, the root growth, and longevity of lucerne plants are generally associated with a high content of carbohydrate and reserve nitrogen in the roots. Susceptibility to winter injury is increased by low percentages of dry matter and low concentrations of carbohydrates and reserve nitrogen in the roots at winter dormancy.

CHEMICAL ABSTRACTS.

Effects due to spraying of fruits. P. R. V. D. R. COPEMAN (S. African J. Sci., 1927, 24, 198—203).—It has been noted that there is a deterioration of flavour in oranges sprayed with lead arsenate. Determinations of acidity, sucrose, and total sugar in the juice showed a marked reduction of acidity in sprayed fruits and a diminution in the ratio of sucrose to total sugar (presumably due to the conversion of sucrose into invert sugar), though the total sugar content is not affected. It is considered that the decrease of both acidity and sucrose accounts for the development of the insipid flavour. The action appears to be confined to arsenical sprays. A similar reduction of acidity occurs in apples and pears sprayed with lead arsenate, but the magnitude of the change is much less than in the case of oranges, and is not sufficient to alter the flavour.

C. T. GIMMINGHAM.

Influence of fertiliser treatments on maturity and yield of cotton. J. W. MUSGRAVE and D. C. COE (J. Amer. Soc. Agron., 1927, 19, 910—923).—High percentages of nitrogen delay maturity. The yields are larger when the fertiliser is applied before planting.

CHEMICAL ABSTRACTS.

Burnt limestones in relation to quality of Bordeaux mixtures. R. H. CARR and L. N. BEMILLER (Ind. Eng. Chem., 1928, 20, 514—516).—Experiments with limestones of known composition and with mixtures of pure calcium and magnesium oxides showed that the proportions of the two oxides in the lime used in preparing Bordeaux mixtures have a marked and consistent effect on the alkalinity of the liquid above the precipitate, on the colour and rate of subsidence of the precipitate, and on the length of time the mixture will retain its colloidal nature and be suitable for spraying. Alkalinity of the liquid decreased as the proportion of magnesium oxide was increased, and characteristic changes in the colloidal state of the precipitate were also noted. C. T. GIMMINGHAM.

Manufacture of manure from straw and calcium cyanamide. F. ZUCKER (Z. Pflanz. Düng., 1928, 7B, 206—215).—Calcium cyanamide was used successfully in making artificial farmyard manure from straw. The best results were obtained when the heaps of straw were about 90 cm. in height. Careful control of moisture conditions was necessary to ensure complete rotting through the stack with minimum losses in dry weight. Pot experiments served to show the value of the manure, but, in general, this was somewhat below that of ordinary yard manure. A. G. POLLARD.

Liquid manure, urine, and drainage water. STIEHR and M. BERGOLD (Z. Pflanz. Düng., 1928, 7B, 216—226).—The addition to cattle urine of sulphuric or phosphoric acid in amounts corresponding to its total nitrogen content reduces nitrogen losses by checking fermentation. The loss of nitrogen during the fermentation of liquid manure is traced almost entirely to the volatilisation of ammonia, no free nitrogen being liberated. Drainage from "hot fermented" manure (Krantz) differs from ordinary liquid manure in that a large proportion of its total nitrogen is in organic combination. It is also richer in potash and phosphate, the latter existing mainly in colloidal suspension. A. G. POLLARD.

Utilisation of waste water from sugar factories. M. GREVEMEYER (Z. Ver. Deut. Zucker-Ind., 1928, 135—139).—Pots containing the following materials were sown with mustard: (a) loamy soil, (b) similar soil saturated with waste water, (c) soil with lime sludge, (d) soil with nitrogen as ammonium sulphate and lime as chalk to correspond with sludge, and (e) completely manured soil. The fresh weights of the crops (in grams) were (a) 128.5, (b) plants all died down, (c) 132.1, (d) 224.5, (e) 265.2. Oats sowed subsequently in the same pots yielded 38, 252.5, 45, 45.5, and 46 g. as fresh plant weight respectively. The failure of mustard in (b) is attributed to the possible presence of harmful salts and the slimy condition of the soil. In the case of oats, soil (b) was less exhausted by the previous crop, and nitrogenous substances may have broken down to a more available form. To other series of pots soil which had been irrigated by waste water was applied as manure at a rate corresponding to 10 tons/acre. The materials used, when extracted with 10% hydrochloric acid, yielded 0.109—0.299% K₂O, 0.305—0.484% N, 0.038—0.332% P₂O₅, 0.70—0.84% CaO, 0.054—0.123% MgO, and with barley gave increased yields of about 10%. With mustard the increase was less marked.

F. E. DAY.

Artificial farmyard manure. M. CARBERY and R. S. FINLOW (Agric. J. India, 1928, 23, 80—85).—The successful preparation of artificial farmyard manure from sugar-cane waste, straw, weeds, etc. is described. Heaps of the material were wetted with diluted cattle urine and sprinkled with bone meal. Better results were thus obtained than when ammonium acetate was employed as a "starter." Field trials demonstrated the satisfactory nature of the material. A. G. POLLARD.

Insecticidal action of esters of halogenated fatty acids in the vapour phase. R. C. ROARK and R. T. COTTON (Ind. Eng. Chem., 1928, 20, 512—514).—The

minimum lethal dose for the rice weevil of various esters of chloroformic, monochloro-, dichloro-, and monobromo-acetic, and chloro- and bromo-propionic acids in the vapour phase has been determined under laboratory conditions. The monochloroacetates appeared to possess interest as possible fumigants and were tested in a fumigation vault on a large scale. The methyl compound was effective against various stored-product insects at the rate of 1 lb. per 1000 cub. ft., and the ethyl compound at 2 lb. at a temperature of 27° or above. The monochloroacetates are, however, injurious to seed wheat.

C. T. GIMINGHAM.

Electrolytic apparatus for determination of copper in insecticides and fungicides. E. B. HOLLAND and G. M. GILLIGAN (*Ind. Eng. Chem.*, 1928, 20, 533).—A form of apparatus with fixed electrodes is described and figured.

C. T. GIMINGHAM.

Feeding of sorghum silage and concentrate to Scindi calves. F. J. WARTH and S. K. MISRA (*Mem. Dep. Agric. India, Chem. Series*, 1927, 9, 125—153).

PATENTS.

Treatment of phosphates or phosphate-containing substances decomposed by sulphuric acid. STOCKHOLMS SUPERFOSFAT FABR. AKTIEBOLAG (B.P. 264,867, 21.1.27. Swed., 23.1.26).—The product obtained by decomposition with sulphuric acid of the phosphate or phosphatic substances, which may be of low grade with high content of iron, alumina, or carbonates, is treated in suspension with milk of lime or limestone powder, and then stirred at about 40° for not more than 6 hrs. with ammonium carbonate or ammonia and carbon dioxide, a saturated solution of ammonium sulphate being used as a carrier for the latter reagents. In this way di- and tri-calcium phosphates soluble in citrate or citric acid solutions are precipitated, and the sulphuric acid used for the decomposition is converted into ammonium sulphate. The liquid is heated to dissolve the ammonium sulphate, the calcium salts are separated by filtration, and the ammonium sulphate formed during the process is recovered by cooling the filtrate.

F. R. ENNOS.

Manufacture of fertilisers. D. TYRER (B.P. 290,075, 2.8.27).—The potassium sulphide present in the product obtained by heating a mixture of calcium phosphate and potassium sulphate or bisulphate under reducing conditions is decomposed by treatment with an acid or acid salt containing an acid radical of fertilising value, e.g., phosphoric acid or nitric acid, and, if desired, the acid salts in the product are neutralised with ammonia.

L. A. COLES.

Manufacture of insecticides, disinfectants, and fertilisers. V. CASABURI (B.P. 289,498, 24.11.26).—A process for the manufacture of solutions, powders, and semi-solid mixtures of various known insecticides or disinfecting agents with hemicelluloses obtained from locust-tree beans is claimed. The hemicelluloses are colloidal and have high adhesive powers. Ground fertilisers may be added to the mixtures.

C. T. GIMINGHAM.

XVII.—SUGARS; STARCHES; GUMS.

Correct procedure in sulphitation of thin [sugar] juice. I. Sulphitation. E. THIELEPAPE and P. MEIER (*Z. Ver. deut. Zucker-Ind.*, 1928, 233—241).—In order to test the relative merits of sulphitation before the second carbonatation and of simultaneous treatment with sulphur dioxide and carbon dioxide, the amount of sulphite remaining in solution in either case has been studied. Solutions of lime, with and without sugar, and limed juices, were treated with known amounts of sulphur dioxide and carbon dioxide by both methods. Successive treatment removed a distinctly larger amount of sulphite than simultaneous treatment.

F. E. DAY.

Gilchrist process of [sugar juice] clarification. J. J. SEIF (*Internat. Sugar J.*, 1928, 30, 82—84).—Some advantages of the Gilchrist process (in which the juice is limed while hot with calcium saccharate under definite hydrogen-ion control; cf. B.P. 247,542; B., 1927, 952) as compared with ordinary liming are: a more intense flocculation with inclusion of fine particles, a better elimination of colloids, a lighter coloured juice, less lime consumption, and, in general, a better control and more rapid operation. The mud from the settler has pH 6.9—7.3, which is considered ideal for filter-press work in giving a well-exhausted cake.

J. P. OGILVIE.

Factors influencing the filtration of raw sugar solutions. R. H. KING (*The Planter*, 1927, 79, 221—223, 242—244, 264—267).—A readily filtering sugar demands good clarification in its manufacture, the phosphoric acid content of the juice being the major factor in securing this. Suspensions which impede filtration are the result of poor defecation, solubility of certain substances which are precipitated by concentration in the clarified juice, redissolution of the settlings during washing, and also the introduction of insoluble matter from the low-grade sugar re-melted. Suspensions capable of forming precipitates that retard filtration are formed as the result of the digestion of fine particles of bagasse during treatment with lime at a high temperature.

J. P. OGILVIE.

Bleaching of off-colour sugar crystals. J. P. OGILVIE and R. G. W. FARNELL (*Internat. Sugar J.*, 1928, 30, 92—93).—Experiments on the effect of various gases on "off-colour" sugars showed a distinct improvement in the case of beet sugars with chlorine and ozone, but with cane crystals none of the gases applied (which also included sulphur dioxide and oxides of nitrogen) effected any noteworthy bleaching, due possibly to the fact that the discoloration of this class of sugar is due largely to insoluble tannin matter.

J. P. OGILVIE.

Value of balancing the total soluble solids of the cane sugar factory. E. E. BATTELLE (*Internat. Sugar J.*, 1928, 30, 89—91).—In appraising the relative technical efficiency of several cane factories, in addition to the usual balance-sheet relating to the polarisation of the various products, it is advantageous to extend this to include a statement of the total soluble solids as well. By this means losses are often disclosed which may not be apparent in the present scheme of reporting.

J. P. OGILVIE.

Treatment of effluents from beet sugar factories.

B. J. OWEN (Internat. Sugar J., 1928, 30, 75—79).—A Brackett 2-stage, cup-type screen placed beneath the outlet of the washer was found to remove the whole of the rootlets, leaves, small gravel, and earth, and subsequently a Wyllie gravel filter left the effluent clear and bright. It is concluded that the problem of dealing with conveying and washing waters seems capable of easy solution by this method, as they can be discharged direct to the river or recirculated. J. P. OGILVIE.

Nature of the neutralisation precipitate and its effect on the recovery of milk sugar [lactose] from grain-curd casein whey. R. W. BELL and P. N. PETER (Ind. Eng. Chem., 1928, 20, 510—512).—Acid wheys, such as grain-curd casein whey, prepared by coagulation with dilute acid, contain nearly all the salts of milk and on neutralisation yield a precipitate of calcium phosphate and protein material, which increases the viscosity of the concentrated whey and prevents crystallisation of the lactose. The quantity of this precipitate and the ratio of calcium phosphate to protein material increases with a decrease in the titratable acidity of the liquid. The complete removal of this precipitate is an essential to the successful recovery of lactose from the acid whey. F. R. ENNOS.

Yeast gum. Y. HASHITANI (Bull. Agric. Chem. Soc. Japan, 1927, 3, 2—14; cf. B., 1927, 567).—Yeast gum forms a *benzoyl* derivative, $C_6H_5O_5Bz_2$, m.p. 223—225°, decomp. 240°, and an *acetyl* derivative, $C_6H_7O_5Ac_3$, m.p. 178—182°; depolymerisation of yeast gum with glycerol at 210—220° yields α -yeast gum, $[\alpha]_D^{20} +80.08^\circ$, mol. wt. 808 (*dibenzoyl* derivative, m.p. 198—199°; *triacetyl* derivative, m.p. 158—159°).

CHEMICAL ABSTRACTS.

Sugar factory waste waters. GREVEMEYER.—See XVI. **Glucose.** MEZZADROLI.—See XVIII.

PATENT.

Clarification of liquids [sugar juices]. A. VALLÉE y GUMÁ (U.S.P. 1,665,167, 3.4.28. Appl., 7.11.21. Cuba, 21.10.21).—Sugar juices, after the usual liming, are boiled vigorously to remove air and vapours. The subsequent clarification by settling is facilitated by maintaining the temperature at 100°. Other liquids, boiling above 100°, may also be kept, during clarification, at those temperatures at which the differences between the densities of the liquids and their suspended solids are greatest. F. G. CLARKE.

XVIII.—FERMENTATION INDUSTRIES.

Taka-invertase. II. R. WEIDENHAGEN and B. B. DEY (Z. Ver. Dent. Zucker-Ind., 1928, 242—261; cf. B., 1928, 423).—Comparison of the rates of inversion of sucrose by taka-invertase and by invertase from a top-fermentation yeast showed that as the reaction proceeded the velocity coefficient decreased in the former, but increased in the latter case. These departures from the unimolecular form of the reaction may be characteristic of invertases from the two sources. Contrary to the statement of Leibowitz and Mechlinski (A., 1926,

865), taka-invertase hydrolysed raffinose more than twice as fast as did yeast invertase of a similar sucrose-inverting power. This reaction closely followed the unimolecular form. The action of taka-invertase on sucrose and raffinose was markedly inhibited by α -glucose, less by β -glucose, and only slightly by *lævulose*; in fact, *lævulose* was without influence in the case of sucrose. The action of yeast invertase was most strongly inhibited by β -glucose; α -glucose had least effect on its hydrolysing action on sucrose, *lævulose* least on its action on raffinose. Since the inhibiting effect of the hexose was found to increase with decreasing amounts of substrate, it is concluded that the effect is due to affinity between hexose and enzyme.

F. E. DAY.

Comparison of acid production of *B. Delbrücki* and the cold lactic acid bacteria (*B. lactis acidii* and *B. cucumeris fermentati*, Henneberg). STAIGER and M. GLAUBITZ (Z. Spiritusind., 1928, 51, 109—110).—Solutions of sugars of 5% and 10% concentration, containing 1.5% and 3% of yeast extract, respectively, were employed. Molasses of 10% and 20% concentration, equivalent to 5% and 10% of sucrose, were also used, with and without yeast extract. One set was inoculated with *B. Delbrücki* and incubated at 45° for 18 days, the other with equal amounts of *B. lactis acidii* and *B. cucumeris fermentati* and kept at room temperature for a similar period. 250 c.c. of each culture medium were used, and the two kinds of bacteria gave the following amounts (c.c.) of *N*-lactic acid, respectively. In sucrose 5%, 17.5, 26.25; 10%, 21.25, 36.25; dextrose 5%, 8.75, 23.75; 10%, 10.00, 33.75; maltose 5%, 12.25, 28.75; 10%, 12.25, 35.00; molasses and yeast extract 10%, 28.75, 45.00; 20%, 35.75, 57.50; molasses without yeast extract 10%, 11.25, 18.75; 20%, 12.50, 20.00. F. E. DAY.

Saccharification of amylaceous substances and manufacture of ethyl alcohol and dextrose in an antiseptic medium by means of oriental bio-enzymes. G. MEZZADROLI (Zymologica, 1928, 3, 37—39).—If a sample removed from an aseptic saccharification vessel is passed through a filter and the filtrate, treated with a few drops of toluene, xylene, thymol, formaldehyde, fluoride, etc., is kept at the same temperature as the original vessel, the formation of sugar proceeds *pari passu* with that in the vessel. The mucor (*amylo*) process may, therefore, be modified as follows: The grain, broken into three or four, is placed in a bath at 50—60° containing about 8 pts. per 1000 of hydrochloric acid (*d* 1.16), left for about 2 hrs., and heated in a cooker, preferably horizontal, for 40—60 min. at 4 kg. pressure. When the liquor begins to filter well through filter-paper, the mass is discharged into a closed, aseptic vessel, cooled and aerated with sterile air, and at 40° seeded with a flask of sporulating mucor 105. After 15—18 hrs. from the time of seeding the mycelia begin to appear and soon assume their normal appearance, and when saccharification has produced 30—50 g. of sugar per litre, toluene or fluoride is added in sufficient amount and the mash is discharged into open vats. When the sugar reaches 50—80 g./litre, yeast acclimatised to the antiseptic is introduced. The claims for this procedure

are cheaper plant, lower coal consumption, acceleration of the various operations, and diminished chances of failure.

T. H. POPE.

Electrocolorimeter for the photo-electric measurement of malt colours. F. STOCKHAUSEN and F. WINDISCH (Woch. Brau., 1928, 45, 231—232).—The light from a small lamp passes through a cell containing the malt extract to be examined, and impinges on a potassium photo-electric cell. No details are given of the means by which the current from this is magnified to operate the pointer-type voltmeter on which the colour is read directly. The scale zero and light intensity are adjusted by two variable rheostats. Determination of colour can be carried out in a few seconds.

F. E. DAY.

Linear or logarithmic calculation of colour in malt analysis. BERMAN and L. LAUFER (Woch. Brau., 1928, 45, 183). H. K. SCHINDLER (*Ibid.*, 183—185). F. DUCHÁČEK and V. L. ŽILA (*Ibid.*, 236—238). F. KUTTER (*Ibid.*, 238—239).—It is proposed that in calculating the observed colour of a laboratory wort of about 8.5% concentration to that of the 10% wort, the proportion should be $\log 10 / \log 8.5$ instead of $10/8.5$. An actual example is given in which the observed colours were: 8.6% 0.16; 9.95% 0.175. The linear calculation from the former to the latter gave 0.185, the logarithmic 0.171.

A reply to Bermann and Laufer (preceding abstract). From a mathematical consideration of the Weber-Fechner law it is concluded that the linear relationship should be used in the calculation. Actual examples of wort prepared at different concentrations are: Concentration 8.40%, colour (observed) 0.20; concentration 9.95%, colour (observed) 0.24, by calculation (linear) 0.237, (logarithmic) 0.216; calculating from strong to weak wort, concentration 8.35%, colour (observed) 0.46; concentration 4.175%, colour (observed) 0.24, by calculation (linear) 0.23, (logarithmic) 0.31. The case quoted by Bermann and Laufer shows too little difference to be significant.

The correctness of the linear calculation on the basis of the Lambert-Beer law of absorption is discussed, and its practical accuracy verified by colour determinations on worts prepared at various concentrations from the same malts.

Extracts of varying concentration from the same malt had colours which were approximately in direct linear proportion to the concentration. Though the linear calculation is only approximate, the logarithmic calculation is quite erroneous.

F. E. DAY.

Simple graphical method for determining the course of the natural distillation process. H. BRANDES (Chem. Fabr., 1928, 261—262).—The construction of a curve showing the relation between the percentages of alcohol in the vapour and in the residual liquid in the flask during the course of distilling a mixture of alcohol and water without the aid of a fractionating column is described, and a geometrical construction is given for deriving from this curve a curve showing the percentage of alcohol in the residue at any stage of the distillation.

A. R. POWELL.

Yeast gum. HASHITANI.—See XVII. **Essential oil of hops.** CHAPMAN.—See XX.

PATENTS.

Manufacture of beverages. H. MACKESON (B.P. 289,601, 9.3.27. Addn. to B.P. 267,236; B., 1927, 375).—The addition either of the priming solution containing malt extract after fermentation or of the malt extract before fermentation is omitted.

F. R. ENNOS.

Manufacture of a dry urease preparation. HENKEL & Co. G.M.B.H. (B.P. 277,644, 30.8.27. Ger., 15.9.26).—A stable, soluble urease preparation is obtained by adding substances, such as phosphates to a filtered or centrifuged aqueous extract of soya or jack beans or similar urease-containing parts of plants, and then atomising the solution in a dry, heated current of air or gas; or the extraction may be made with solutions of the phosphates etc. above-mentioned.

B. FULLMAN.

Dehydration of alcohol. J. A. STEFFENS (U.S.P. 1,670,053, 15.5.28. Appl., 16.8.23).—See B.P. 220,606; B., 1925, 185.

Fermenting products (B.P. 289,978).—See XIX.

XIX.—FOODS.

Action of cold on the fats of milk. G. QUAGLIA-RIELLO (Atti R. Accad. Lincei, 1928, [vi], 7, 75—78).—The lowering of the surface tension occurring when milk is cooled for 30 min. at below 10° is due to passage of triglycerides of the lower fatty acids from the fat globules to the interglobular liquid, in which they dissolve. The phenomenon is not shown by homogenised milk.

T. H. POPE.

Grading of commercial gelatin and its use in the manufacture of ice cream. II. A. C. DAHLBERG, D. C. CARPENTER, and J. C. HENING (Ind. Eng. Chem., 1928, 20, 516—526).—Methods used for grading gelatins, both commercial and high-grade varieties, are compared as to their suitability for the manufacture of ice cream. In weak solutions a reduction in the ageing temperature and the presence of the serum solids of milk caused an increase in the extent of gelation. The minimum percentages of each gelatin required to give the desired body and texture to the ice cream, as well as the requisite character after melting, were also determined, and a simple practical test is described for this purpose. Provided the correct amount was used, the quality and volume yield of ice cream did not appear to be affected by the kind or strength of the gelatin. The principal property of gelatin which is of value in ice cream manufacture appears to be its ability to re-form a gel at low temperatures after being whipped to a fluid condition; the agar gels do not behave in this way.

F. R. ENNOS.

Adsorption phenomena [with wheat]. G. TESTONI (Rev. gén. Colloid., 1928, 6, 8—15).—In an attempt to decide whether gluten is already existent in wheat flour, or whether it is produced during the mixing with water, the author has studied the colours produced in a

wheat grain by adsorption of various natural and artificial organic colouring matters. It is concluded that gluten is not pre-existent in wheat flour, but is formed under the influence of calcium salts, when mixed with water. The sensitivity of indicators is diminished by adsorption of the indicator at a surface, and measurement of the decrease in sensitivity provides a means of characterising the adsorbent surface. The values are expressed as "adsorption values," and some of the proteins of wheat have been characterised in this way. Ferric and cupric salts are adsorbed by gluten and gliadin, and the colourless products fail to give reactions for the respective metal ions except towards potassium ferrocyanide. The product of adsorption of nickel salts fails to give a reaction for nickel ions with any reagent. It is suggested that reaction can take place only when the second substance also is adsorbed, and that this condition is realised in the case of potassium ferrocyanide.

E. S. HEDGES.

Thermophilic and thermoduric micro-organisms, with special reference to species isolated from milk. II. Thermal resistance of micro-organisms. A. H. ROBERTSON (Vt. Agric. Exp. Sta. Bull., 1927, 274, 1—27).

PATENTS.

Manufacture of fermenting products. BRIT. ARKADY CO., LTD., R. WHYMPER, and H. HEWITT (B.P. 289,978, 12.2.27).—Bread is made by adding to the dough or incorporating with the flour and other ingredients used in making the dough a fermenting product comprising yeast and a fungus such as *Aspergillus oryzae* or *Penicillium*, together with the products obtained by the previous action of either or both of these micro-organisms on rice, barley, or vegetable substances rich in proteins.

C. RANKEN.

Treatment of dried fruit. E. C. R. MARKS. From SUN-MAID RAISIN GROWERS OF CALIFORNIA (B.P. 289,722, 11.11.27).—Raisins are subjected to opposing blasts of highly ozonised air and steam as they fall through a vertical chute. They are then quickly dried at 400° and the heated vapours removed.

F. R. ENNOS.

Preservation of fodder. E. C. R. MARKS. From M. FLUBACHER (B.P. 285,939, 22.10.26).—The fodder plants are cut and packed by pressing in air-tight containers, each layer being subjected to heavy pressure in order to prevent injurious fermentation, self-heating, or breathing of the plants. A 3% solution of a mixture composed of sodium chloride 50%, calcium chloride 10%, sodium phosphate 10%, and ferrous lactate 30% is used to spray the fodder either before or after cutting, or during the packing into the containers, whereby butyric fermentation is prevented.

F. R. ENNOS.

Preparation of a composition for coating substances [foods] liable to putrefaction. G. VAN DER VEEN and G. J. P. H. A. DE BEUS (B.P. 286,096, 14.3.27).—Rosin, purified by fusion with soda, is melted with solid paraffin in such proportion as to reduce the m.p. to 25—30°. Activated charcoal, oxide or carbonate of magnesium, and, if desired, magnesium chloride, may be added to the melted mixture.

F. R. ENNOS.

Sterilisation of clear and turbid liquids [foods] by means of ultra-violet rays. E. O. SCHEIDT (U.S.P. 1,670,217, 15.5.28. Appl., 24.10.25. Ger., 5.9.25).—See B.P. 257,956; B., 1928, 106.

Production of a [grated] almond milk food product. II. HOFER-MASSARD (B.P. 282,088, 9.12.27. Switz., 10.12.26).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Composition of tragacanth-paraffin oil emulsions. E. SCHULEK and G. VASTAGH (Pharm. Zentr., 1928, 69, 275—276).—The emulsion is first separated by warming with alcohol and the paraffin oil is extracted with light petroleum. The petrol extract is evaporated and dried at 120°. The presence of saccharin in the aqueous-alcoholic residue was demonstrated. The results of two analyses are appended.

H. BURTON.

Nicotine in tobacco. II. A. HEIDUSCHKA and F. MUTH (Pharm. Zentr., 1928, 69, 305—307; cf. B., 1927, 570).—Using the methods of Rasmussen (A., 1916, ii, 359) previously employed, and that of Pfyl and Schmitt (B., 1927, 955), determinations were made of the nicotine content of tobacco and smoke from various German cigarettes, including special brands advertised as harmless or poor in nicotine. The presence of nicotine could be confirmed in all cases by determination of the m.p. of the dipicrate. The amount of nicotine found in the tobacco and smoke respectively, calculated in each case on the weight of dry tobacco, was: ordinary cigarettes, 1.11—1.31% and 0.16—0.23%; special brands, 0.64—1.22% and 0.09—0.16%.

R. K. CALLOW.

Determination of alkaloids in ergot. F. WESSEL (Pharm.-Ztg., 1928, 73, 354—355).—The procedure recommended in the German Pharmacopœia, 6th Ed., is very troublesome, emulsions being formed which render filtration difficult; the titration of the alkaloids isolated is also inaccurate. It is better to remove fats by a preliminary extraction with light petroleum, and to precipitate the alkaloids from the hydrochloric acid solution obtained with ammonia, instead of with sodium carbonate. The precipitated alkaloids are dried and weighed.

S. I. LEVY.

Volumetric determination of aluminium in pharmaceutical preparations. H. MATTHES and P. SCHÜTZ (Pharm.-Ztg., 1928, 73, 353—354).—Details of the procedure for the precipitation of the 8-hydroxyquinoline compound of the metal, and the determination of the hydroxyquinoline obtained from the precipitate by treatment with acid, are given. The latter is based on the formation of the dibromo-substitution product by the action of potassium bromide and bromate; the excess of bromate used is determined iodometrically.

S. I. LEVY.

Higher-boiling constituents of essential oil of hops. A. C. CHAPMAN (J.C.S., 1928, 1303—1306).—Distillation of the higher-boiling constituents of hop oil gave seven fractions, b.p. 87—97° to 180—200°/4 mm., d_{20}^{20} 0.8948—0.9560, n_D^{20} 1.4769—1.4975. The first fraction, after hydrolysis with alcoholic potassium

hydroxide and subsequent removal of alcohols with phthalic anhydride, yielded a ketone, $C_{15}H_{22}O$, having b.p. 74–76°/3 mm., d_{20}^{20} 0.8861, n_D^{20} 1.485, $[\alpha]_D^{20}$ –0.4°, when regenerated from its semicarbazone, m.p. 98°. The name *lupayone* is suggested for the ketone. From the fractions of b.p. 110–150°/4 mm., after successive treatment with alcoholic potassium hydroxide and phthalic anhydride, there was obtained an unsaturated alcohol (*luparenol*), $C_{15}H_{24}O$, having one double linking, b.p. 125–128°/3 mm., d_{20}^{20} 0.9738, n_D^{20} 1.5023, $[\alpha]_D^{20}$ –3.7° (phenylcarbimide derivative, m.p. 157°). The residue, when purified by successive treatment with phthalic anhydride and alcoholic silver nitrate, gave a phenolic ether (*luparol*), $C_{16}H_{26}O_2$, b.p. 122–124°/2 mm., d_{20}^{20} 0.9170, n_D^{20} 1.4942. When luparol is boiled with 50% aqueous potassium hydroxide it undergoes gradual decomposition into isovaleric acid, also obtained by oxidation with potassium permanganate, and a phenol, probably $C_{11}H_{16}O_2$, b.p. 115–117°/4 mm., d_{20}^{20} 0.9448, n_D^{20} 1.4670. The fractions of b.p. 150–200°/4 mm. consist chiefly of luparol and luparenol. isovaleric acid is a constituent of the original oil. H. BURTON.

Essential oils of *Eucalyptus micrantha* (D.C.) and *E. haemastoma* (Smith). I. A. R. PENFOLD and F. R. MORRISON (J. Proc. Roy. Soc. New South Wales, 1927, 61, 267–268).—Steam distillation of 6 lots of leaves and terminal branchlets of *E. micrantha* (D.C.) gave yields of oil varying from 0.62 to 0.9% and having d_{15}^{15} 0.8883–0.9122, α_D^{20} –25.5° to +5°, n_D^{20} 1.4701–1.4892, ester value 6.2–23.1, ester value after acetylation 41.9–95.2, solubility in 80% alcohol 1 vol.—8 vols. The principal constituents of the oils were *l*- α -phellandrene (moderate to large quantities), sesquiterpenes, terpineol (?) and piperitol and their hexoic esters, *d*- α -pinene, cineole, sesquiterpene alcohols, and traces of aromatic aldehydes. Piperitone was probably present and eudesmol was present in one oil. Leaves and terminal branchlets of *E. haemastoma* (Smith), collected at the same time and place as those of *E. micrantha*, on steam distillation gave 0.25–0.5% of oil having d_{15}^{15} 0.9295–0.9571, α_D^{20} +3.3° to +16.8°, n_D^{20} 1.4861–1.4957, ester value 8.6–12.0, ester value after acetylation 72.5–116.9, solubility in 70% alcohol 1.7–2.0 vols., and containing eudesmol, sesquiterpenes (aromadendrene and probably eudesmene), *d*- α -pinene, cineole (10–15%), and a very small quantity of phellandrene not detectable by the B.P. test.

E. H. SHARPLES.

Essential oils of two species of *Baeckea*. A. R. PENFOLD (J. Proc. Roy. Soc. New South Wales, 1927, 61, 285–295).—Steam distillation of the leaves and terminal branchlets of 2 lots of *Baeckea brevifolia* (De Candolle) collected in 1925 and 1927, gave 1.6% and 1.02% of oils having the following respective constants, d_{15}^{15} 0.9110, 0.9257; α_D^{20} –3.9°, —; n_D^{20} 1.4839, 1.4888; ester value 3.2, 5.4; ester value after acetylation 75, 109. Both oils were soluble in 0.6 vol. of 80% alcohol and insoluble in 10 vols. of 70% alcohol, and the principal constituents were: α - and β -pinene, the latter predominating, cineole, eudesmol (30–45%) (*allophanate*, m.p. 174°), and small quantities of phenolic substances.

In a similar manner the leaves and terminal branchlets of *Baeckea linifolia* var. *brevifolia* (Mueller) gave 0.5–0.82% of mobile oil having d_{15}^{15} 0.8917–0.9035, α_D^{20} +6.85° to +10.85°, n_D^{20} 1.4752–1.4791, ester value 8.3–21.2, ester value after acetylation 44.7–60.9, solubility in 80% alcohol (by wt.) 4.6–5.0, and consisting of α - and β -pinene, cymene, cineole (18%), sesquiterpene and sesquiterpene alcohol, an unidentified alcohol (d_{15}^{15} 0.915, α_D^{20} +7.3, n_D^{20} 1.4806; naphthylurethane, m.p. 120°), a mixture of phenols belonging to the tasmanol group, and isobutyric and isovaleric acids present as esters.

E. H. SHARPLES.

Essential oil from timber of rosewood (*Dysoxylon Fraserianum*). A. R. PENFOLD (J. Proc. Roy. Soc. New South Wales, 1927, 61, 337–346).—Three separate steam distillations of the shavings of rosewood gave a yield, on the wet wood (41–46.13% moisture), of 1.2–2.7% of pale to dark blue, viscous oil having d_{15}^{15} 0.925–0.9398, α_D^{20} –5°, n_D^{20} 1.5035–1.5046, ester value 2.2–10.6, ester value after acetylation 40.4–62.5, and was insoluble in 10 vols. of 90% alcohol (by wt.). The oil consisted of a mixture of sesquiterpenes, possibly two, which are apparently hydroazulenes, as on dehydrogenation with sulphur azulene was obtained, a third sesquiterpene, *dysoxylonene* (b.p. 136–137°/10 mm., d_{15}^{15} 0.9236, α_D^{20} nil, n_D^{20} 1.5063; *dihydrochloride*, m.p. 108–109°, α_D nil) evidently related to the cadinene group. Cadinene was found to be the principal sesquiterpene in the first two distillations. Azulene, unidentified phenols, and probably copaene and aromadendrene were present in the oils.

E. H. SHARPLES.

Determination of eugenol. W. A. N. MARKWELL and L. J. WALKER (Perf. Ess. Oil Rec., 1928, 19, 169).—For the determination of eugenol in a number of samples of oil the following method is quicker, more convenient, and necessitates a smaller quantity of oil than that in which Hirschsohn flasks are used. 2 c.c. of the oils are measured into each of several Gerber milk tubes containing 16 c.c. of 5% potassium hydroxide solution and the tubes are shaken at 5 min. intervals for 30 min. A further 4 c.c. of the potassium hydroxide solution are added to each and the tubes are centrifuged for 5 min., the pear-shaped bulbs being placed inwards. By adjustment of the stopper the uncombined oil is raised into the graduated stem. The percentage of eugenol is obtained from the formula: % = 100 – 50V/f, where V is the volume of uncombined oil and f is the factor required to convert divisions on the Gerber scale into c.c.

E. H. SHARPLES.

Flour from linseed. ANDRÉ.—See XII.

PATENTS.

Preparation of emetine. E. BURCKHARDT and M. STÄRKLE, Assrs. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,670,059, 15.5.28. Appl., 8.6.27. Switz., 27.5.27).—Cephæline is methylated with phenyltrimethylammonium hydroxide.

C. HOLLINS.

Production of killed hog cholera virus. J. REICHEL (U.S.P. 1,661,233, 6.3.28. Appl., 16.2.25).—Live viru-

lent hog cholera virus in the form of defibrinated blood is treated with 0.5% of phenol, and then kept at 5–10° for 30–90 days, and at about 37° for 30 days. The product is used for immunising hogs against cholera.

B. FULLMAN.

Manufacture of hydrocyclic ω -aminoalkyl compound. H. RUPE, ASSR. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,670,990, 22.5.28. Appl., 2.9.25. Switz., 1.10.24).—See B.P. 240,814; B., 1926, 27.

Manufacture of unsaturated aldehydes. H. RUPE, ASSR. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,670,825, 22.5.28. Appl., 11.3.27. Switz., 22.3.26).—See B.P. 267,954; B., 1927, 571.

Manufacture of basic bismuth salts of aryl-arsinic acids. R. W. E. STICKINGS, ASSR. to MAY & BAKER, LTD. (U.S.P. 1,669,542, 15.5.28. Appl., 2.12.26. U.K., 5.12.25).—See B.P. 266,820; B., 1927, 349.

Treatment of oils (B.P. 281,232).—See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Optical sensitising of silver halide emulsions. I. Adsorption of orthochrome-T to silver bromide. S. E. SHEPPARD and H. CROUCH (J. Physical Chem., 1928, 32, 751–762, and Phot. J., 1928, 68, 273–280).—The partition coefficients for an isocyanine-orthochrome-T bromide between aqueous solutions and chloroform have been measured spectrophotometrically and show higher values in weaker buffer solutions of the same p_H . The partition of the dye in the aqueous layer is lowered by the addition of soluble halides. The absorption spectra of orthochrome-T bromide at p_H 4.8–5.8 show that as the colourless form of the dye increases the ultra-violet absorption increases. In chloroform and in methyl alcohol the absorption maximum is slightly affected. Data on adsorption of the dye at 50° by silver bromide grains are given, and two alternative hypotheses are advanced to explain the results obtained. Experiments on the diffusion of the dye into gelatin cylinders indicated a lowering of diffusivity with rising p_H in agreement with the tendency of the coloured form of the dye to form colloidal micelles of low diffusivity, and, further, that the amount of dye adsorbed at saturation is apparently much greater than the amount taken up by silver halide for optimum sensitising. A mechanism for optical sensitising is suggested.

L. S. THEOBALD.

Photographic prints on fabrics. MICHELS.—See VI.

PATENTS.

Production of photographs in natural colours. W. LANGGUTH and C. HUMMEL (B.P. 274,129, 11.7.27. Ger., 9.7.26).—In the process in which three component pictures are successively printed on the same paper support using azo dyes, the support saturated with the dye components is exposed while moist under the negatives in a cooled frame. The order of printing is blue-green, green-yellow, and finally purple. The separate

component images are printed under filters of colours complementary to the colours being printed. Suitable dye components are, for blue-green: dianisidine and 1:8-aminonaphthol-2:4-disulphonic acid; for the green-yellow: 2-methylbenzidine and ethyl acetoacetate; for the purple picture: *o*-anisidine and β -hydroxynaphthoic acid or its derivatives; in each case the amine is diazotised in presence of sodium sulphite.

W. CLARK.

Production of coloured photographic pictures. J. H. CHRISTENSEN (B.P. 289,175, 26.1.27).—Colour photographs to be viewed by reflected light consist of screen unit colour images of a layer of colour elements covered by a layer containing silver, the film being brought into contact with a coherent white layer reflecting the main part of the light within a depth from its surface not exceeding the average diameter of the coloured elements. Suitable white layers consist of a layer of cellulose derivative penetrated by numerous air-filled fissures, or a cellulose derivative containing a white pigment.

W. CLARK.

Production of photozincograph printing surfaces. F. ULLMANN (B.P. 288,023, 24.3.27).—The zinc plate is treated with the salt of a metal, e.g., aluminium, having a tendency to form double salts, before the application of the sensitive chromate layer. The action between the zinc and the chromate preparations is minimised.

W. CLARK.

XXII.—EXPLOSIVES; MATCHES.

PATENT.

Metal derivatives of diketones (B.P. 289,493).—See III.

XXIII.—SANITATION; WATER PURIFICATION.

Stream-flow sewage-treatment process. H. N. JENKS and M. LEVINE (Eng. News-Rec., 1928, 100, 808–813).—Laboratory and large-scale experiments are described in which the supply of oxygen necessary for the operation of the activated sludge system of sewage purification is obtained by passing the mixed liquor in a shallow stream along an open channel. This is succeeded by a quiescent period in which the oxygen so obtained is utilised biologically in purifying the sewage, which is then again passed over the channel by means of a pump, re-aerated, and the cycle repeated. The frequency of re-aeration can be adjusted so as to conform with the oxygen demand at different stages of the process and thus give increased efficiency. It is claimed that strong domestic sewage can be purified in 6 hrs. with a minimum expenditure of energy for aeration.

C. JEPSON.

Modern aspects of chlorination of water. N. J. HOWARD (J. Amer. Water Works' Assoc., 1928, 19, 546–552).—Recent research suggests that the sterilising action of chlorine is due not to the production of nascent oxygen, but to the formation of some toxic substance which prevents cell division. The substitution of chlorine for alum when the water is physically good results in

considerable saving in the maintenance cost of the filters. A mixture of chlorine and ammonia is considered to possess many advantages over straight chlorine, particularly in a delayed action when dealing with spore-forming bacteria and after-growths in mains. It is said to be tasteless, and in some cases the use of ammonia prior to chlorination has prevented iodoform tastes previously present. This taste is sometimes due to the presence of traces of phenol, cresol, anisole, or xylol. The taste due to chlorine is likely to occur if the amount used is the minimum necessary for sterilisation, but by adding excess of chlorine and giving a certain period of contact before removal of the residual chlorine by sulphur dioxide all taste can be avoided.

C. JEPSON.

Method for phenol determinations [in water]. J. R. BAYLIS (J. Amer. Water Works' Assoc., 1928, 19, 597—604).—Gibbs' method (cf. A., 1927, 688) in which use is made of the blue indophenol colour produced with dibromobenzoquinonechloroimide is suggested. A sample of the water (800 c.c.) is taken and distilled at a rate of 8—10 c.c./min. The distillate is collected in five successive lots of 100 c.c. each, and the phenol determined by comparison of the indophenol colour produced. This amount of distillate contains 85% of the phenol in the original sample. If the total quantity is between 15 and 5 pts./billion it is desirable to concentrate by taking two or more samples each of 800 c.c., distilling over 50% of the sample (containing 75% of the original phenol), and determining the phenol in the combined distillates. It is considered that the smallest quantity of phenol which will cause noticeable chlorophenol tastes is 5 pts./billion. Before adding the dibromobenzoquinonechloroimide, the distillates should be buffered so as to have p_H 9.6—10.0 as this influences the rate of indophenol formation.

C. JEPSON.

Test for phenolic tastes and odours in water after chlorination. F. W. SPERR, JUN., W. H. FULWEILER, F. E. DANIELS, and O. O. MALLEIS (J. Amer. Water Works' Assoc., 1928, 19, 605—606).—A sample (500 c.c.) is made acid to litmus by means of sulphuric acid and distilled. The distillate (250 c.c.) is collected in a 500 c.c. graduated flask, made up to 500 c.c. with distilled water, and diluted as follows: 1 to 10; 1 to 100; 1 to 1000, etc. 200 c.c. of the distillate and a like amount of each dilution are then taken and treated with a slight excess of chlorine water (a total of 0.3 p.p.m. is usually enough). After 15 min. the excess chlorine is boiled off and the odour test made on the hot liquid, after which it is allowed to cool and tasted. Results should be expressed as the lowest dilution in which the taste and odour are negative. C. JEPSON.

Volumetric method for determination of sulphate ion [in potable waters etc.]. F. G. GERMUTH (J. Amer. Water Works' Assoc., 1928, 19, 607—609).—For determining sulphate present in potable waters or other materials, in which the amount present is small, a slight excess of 0.02N-barium chloride is added, with vigorous stirring, to a measured volume of sample slightly acidified with hydrochloric acid and at room temperature. After 5 min. it is made alkaline to phenol-

phthalein by means of ammonia, and 10 c.c. of 5% ammonium acetate are added. The excess barium chloride is titrated back with 0.02N-potassium chromate, using lead nitrate as an outside indicator, and the amount of sulphate present is calculated from the volume of barium chloride used. C. JEPSON.

Fumigation tests with ethylene dichloride-carbon tetrachloride mixture. L. F. HOYT (Ind. Eng. Chem., 1928, 20, 460—461).—The new fumigating mixture of 3 vols. of ethylene dichloride and 1 vol. of carbon tetrachloride proposed by Cotton and Roark (cf. B., 1927, 862) gave a complete kill of all insects present (meal-moth and clothes-moth larvæ, and flour beetles) when used in a 500 cub. ft. vault at the rate of 14 lb. per 1000 cub. ft. for 24 hrs. at 28°. In an 8000 cub. ft. room the results were also satisfactory, though the insects were killed less rapidly. The mixture is easy to use and appears to be a valuable and safe fumigant.

C. T. GINNINGHAM.

Natural separation of salts from rivers charged with [magnesia-containing] effluent from potash works. EMMERLING (Chem.-Ztg., 1928, 52, 398—399).—The river Wipper, which is charged with magnesia derived from potash works, was found during a part of its course, where it passed through a limestone formation, to deposit a mud containing up to 3% MgO as against 0.4% higher up. The limestone itself was non-magnesian, and any reaction between calcium carbonate and magnesium chloride was out of the question. The limestone is, however, argillaceous, and a suspension of the finely-powdered material in water when shaken with magnesium chloride solution deposited a flocculent precipitate containing magnesia. Gelatinous silicic acid is without effect, and the presence of lime is necessary. It was also found that humic acids precipitate magnesia more readily than lime, and water plants take it up in relatively large quantities from waters containing it.

C. IRWIN.

Colliery waters. SIMPKIN.—See II. **Halogenated fatty acids as insecticides.** ROARK and COTTON.—See XVI. **Effluents from beet sugar factories.** OWEN.—See XVII.

PATENTS.

Softening and purification of water. HÖGANÄS-BILLESCHOLMS AKTIEBOLAG and NORDISKE NATROLITH A./S. (B.P. 279,788, 6.7.27. Swed., 29.10.26).—A base-exchange, water-softening material, depending on the action of siliceous gels, may be produced by calcining "Keuper" clay at not exceeding 800° and subsequently crushing it to the desired size. If the calcination has been carried too far the activity of the gels may be re-established by boiling with a solution of an alkali salt, e.g., sodium chloride. After being used for softening water, the effectiveness of the clay may be regenerated by treatment with hydrochloric acid.

C. JEPSON.

Water-softening filters, and regeneration and purification of same. C. HUFSCHMIDT (B.P. 290,064, 5.7.27).

Slag-sand from refuse (B.P. 280,902).—See IX. **Disinfectants** (B.P. 289,498).—See XVI.

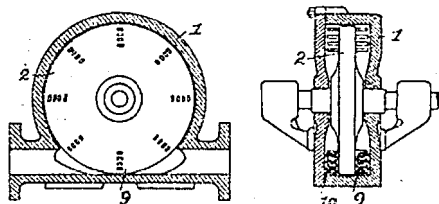
BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

JULY 20, 1928.

I.—GENERAL; PLANT; MACHINERY.

New colloid mill. O. AUSPITZER (Ind. Eng. Chem., 1928, 20, 413—415).—The material to be milled is premixed in a tank fitted with an agitator, and is conducted through the mill chamber tangentially to the beaters as shown in the figure. The exit pipe follows the line which would be naturally taken by the material, so that friction losses, both within and without the mill



chamber, are minimised, and the power consumption is only one seventh of that of a Plauson mill for equal duty. For any given material there is a critical speed of rotation (6000—9000 r.p.m.) of the beaters which is requisite for economical work. The mill is suitable for increasing the covering power of red lead, makes kaolin available for dusting purposes in the rubber industry, and facilitates the preparation of colloidal sulphur, arsenate solutions, etc.

C. IRWIN.

Insulation against heat and cold. I. S. CAMMERER (Chem. Fabr., 1928, 318—320).—Estimates of thermal losses from steam pipes etc. with and without insulation are given. All insulating materials depend for their properties on a porous structure with air spaces, but if such spaces are not quite small convection currents are set up. Qualities desirable for insulating materials for various purposes are described. Whilst a variety of inorganic and organic materials are used for heat insulation, only cork and peat have hitherto found much application for cold-storage insulation. Practical tests are particularly valuable in choosing an insulating material.

C. IRWIN.

Modern methods of insulation. I. S. CAMMERER (Chem. Fabr., 1928, 341—342).—Approximate data are given for the economical degree of insulation for varying pipe diameters and temperature differences. For the accurate determination of the efficiency of insulating materials the Schmidt method employs an auxiliary surface consisting of a rubber sheet of given dimensions provided on both surfaces with a large number of thermo-elements. This is laid on one side of the material to be tested. A recording millivoltmeter is of great assistance, providing a continuous record of heat loss.

C. IRWIN.

What constitutes an adequate sample? J. C. MUNCH and G. L. BIDWELL (J. Assoc. Off. Agric. Chem., 1928, 11, 220—222).—In the sampling of material such as flour or feed, which consists of a number of separate lots, the number of portions to be taken and mixed to give an adequate sample should be equal to the square root of the total number of lots. T. M. A. TUDHOPE.

Apparatus for measuring hydrogen-ion concentrations. P. HANSEN (Dansk Tidsskr. Farm., 1928, 2, 139—140).—The principle of the apparatus (Wulff's strip colorimeter) depends on the fact that hydrogen and hydroxyl ions diffuse much more rapidly into a colloidal membrane when this is placed in a solution of definite p_H value than an organic dyestuff of high mol. wt. with which the membrane is impregnated will diffuse outwards into the solution. The solution to be tested is placed in a glass vessel and a strip of membrane coloured with a suitable indicator immersed in it. After a few minutes the strip is removed, washed with distilled water, dried with filter paper, and placed in a small frame like the cursor of a slide rule. This is slid over a series of prepared coloured transparent strips of membrane until the colour is matched, when viewed by transmitted light. The intervals between the strips correspond to a difference of 0.2 in p_H value, so that an accuracy of 0.1 is obtainable. Experiments carried out on the determination of the p_H values of solutions by means of the above apparatus gave results which agreed to 0.1 with those obtained by the electrometric method. An advantage of the method over the usual colorimetric ones lies in the fact that it can be employed with coloured or turbid solutions. H. F. HARWOOD.

PATENTS.

Furnaces. G. A. KOHOUT (B.P. 290,027, 20.4.27).—Fuel containing particles of different sizes (e.g., screenings) is charged over a shelf above a blast of air and/or steam which distributes the fuel over a firegrate below, according to the size of the particles, the largest lumps forming the thickest layer near the front; the smaller lumps pass to the back, and the dust does not settle at all, but is burned in suspension. Means are provided (e.g., an inclined grate with rocking firebars) by which the fuel and ash are fed towards the back of the firegrate at such a rate that ash only is left on arrival at the dumping plate at the extreme back.

B. M. VENABLES.

Rotary furnaces. G. BOJNER and A. H. PETERSON (B.P. 269,892, 12.4.27. Swed., 24.4.26).—A rotary furnace is provided with longitudinal gas passages with outlets to the interior of the furnace, and those passages

which are covered by material under treatment are supplied with gas by means which are not described. The passages are formed by overlapping louvre-like members so arranged that the material tends to fall out of them.

B. M. VENABLES.

Furnace. C. V. A. ELEY (B.P. 285,126, 9.11.26).—Air is blown through a number of troughs or tubes with openings in the upper side arranged under the fire. Linked fire bars or a chain grate travel over the troughs and are supported by them on the horizontal inward run. The ash drops off the chain grate at the inner end and falls through a pair of rollers or swinging jaws (by which it is crushed) into a helical conveyor that brings it back to the firing end below the other parts of the apparatus.

B. M. VENABLES.

Furnace and kiln. F. NOVELLI (B.P. 271,889, 26.5.27. It., 27.5.26).—A Hoffmann or other form of kiln for burning bricks, lime, etc. is fired by solid fuel which is charged into vertical retorts within the kiln. The retorts have holes in their walls through which the distillation products pass into the kiln and are there burnt. The coke may either be left in the retorts and removed therefrom as such after the discharging of the kiln, or it may be burnt *in situ* by means of air entering through the holes.

B. M. VENABLES.

Heat exchanger. C. H. POTTS (B.P. 285,524, 8.10.26).—A form of construction of heat exchanger or radiator in which one fluid flows in zig-zag fashion through light, thin, metal elements individually very narrow in one dimension.

B. M. VENABLES.

Incinerators. G. WATSON (B.P. 289,948, 3.2.27).—A boiler occupies one corner of the combustion space of an incinerator and the flue for products of combustion is inside the boiler.

B. M. VENABLES.

Ovens for chemical, metallurgical, and like processes. R. HADDAN. From STETTINER CHAMOTTE-FABR. A.-G. (B.P. 289,128, 17.1.27).—The material is heated while sliding down a sloping hearth, and melts in a chamber at the lower end. Any combustible gases given off during treatment are burnt over the charge or in a chamber underneath the sloping hearth.

B. M. VENABLES.

Crushing or pulverising mills. W. R. HUME (B.P. 289,721, 10.11.27).—A roller works in conjunction with another crushing member (which may also be a roller) and besides its rotation it is caused to have a reciprocating motion towards and from the other crushing member. The reciprocation may be produced by an eccentric roller pressing against the idle side of the crushing roller, the two being held together by springs.

B. M. VENABLES.

Grinding mill. R. SHEPHEARD (U.S.P. 1,659,668, 21.2.28. Appl., 7.3.26).—A rotary shaft extends through a housing provided at one end with means for supplying the material to be ground, and at the other end with means for discharging the ground material. Discs rotating with the shaft have radial slots enabling them to move outwards against the wall of the housing under the centrifugal action. The discs are arranged in pairs, those of each pair being of complementary shape and so nested together that their peripheries travel in the same circumferential path.

H. HOLMES.

Mixing and crushing machine. C. R. DIMM, Assr. to ROBINSON MANUF. CO. (U.S.P. 1,660,221, 21.2.28. Appl., 26.5.26).—A cylinder having an inlet and an outlet both spaced from its ends is provided with a rotary mixing device. Crushing devices are arranged at different angles to the mixing device throughout its length, their side portions being in operable overlapping relation along the cylinder wall.

H. HOLMES.

Production of finely-distributed mixtures of mutually insoluble bodies. M. POLANYI and S. VON BOGDANDY (B.P. 269,586, 14.4.27. Ger., 16.4.26).—The substances to be mixed are vaporised (or cathodically atomised) and then precipitated on a cooled surface; they may be mixed before precipitation, or the effect of rapid alternate precipitations may be produced by allowing the constituents to impinge at different radii on a rapidly rotating cooled drum from which the solid mixture is removed by centrifugal force or scrapers.

B. M. VENABLES.

Apparatus for determining the degree of fineness of pulverulent or fine granular masses by means of suspension. H. HARKORT (U.S.P. 1,667,783, 1.5.28. Appl., 8.12.26. Ger., 14.10.26).—A conical elutriating vessel is provided with a constant-level washing liquid funnel to which are attached easily changed nozzles extending to near the bottom of the elutriating funnel. Adjustments are made by the height and diameter of the nozzles.

B. M. VENABLES.

Drying apparatus. KILMARNOCK ENGINEERING CO., LTD., W. SHAW, and J. S. WILLIAMSON (B.P. 289,655, 26.5.27).—The material passes in succession through a number (say three) of superposed rotating drums, and drying or cooling gases pass through the drums simultaneously. The outlet end of each drum makes a running joint with a female hopper which is extended downwards as a male feed-chute for the next drum below, and the material is caused to accumulate in the hopper and chute to prevent passage of gas therethrough. By this means separate streams of gases may be used, e.g., hot drying gases in the two upper drums and cooling gases in the lowest.

B. M. VENABLES.

Humidification of materials. INDUSTRIAL DRYER CORP., Assees. of G. D. HARRIS (B.P. 264,538, 17.1.27. U.S., 15.1.26).—An unchanged supply of air is circulated over the goods (e.g., leather) through a heater, over a large surface kept moist with cold water, through a fan, and over the goods again. The cold, wet surface is formed by a water-tank below the goods chamber with wicks depending therein.

B. M. VENABLES.

Apparatus for filtering. E. J. SWEETLAND (U.S.P. 1,670,319, 22.5.28. Appl., 10.5.22).—Horizontal, circular filter-leaves are assembled about a central, vertical, perforated sleeve, the perforations communicating with the edges of the filter-leaves. Within the sleeve is a hollow perforated shaft on which it is removably mounted.

F. G. CLARKE.

Continuous filtering apparatus. G. GRÖNDAL (U.S.P. 1,668,557, 8.5.28. Appl., 7.6.23. Swed., 23.4.21).—A number of vacuum filtering elements are arranged and charged similarly to the buckets of an overshot water-wheel, but they are kept horizontal

except for a period on the upward journey, when they are overturned to discharge the filter cakes.

B. M. VENABLES.

Filters for air or gases. A. C. HANDLEY (B.P. 285,561, 17.11.26).—A number of openwork holders are removably supported in a frame. Each holder is charged with coir fibre and may, if desired, be covered with coconut matting. Means are provided for moistening the fibre.

B. M. VENABLES.

Cake-washing means for rotary drum filters. DUNCAN STEWART & Co., LTD. From W. MAUSS (B.P. 284,960, 24.9.27).—On the outgoing side of the drum, partly submerged in the prefilter, is resiliently pressed a segmental shield or container closed at the ends but open at top and bottom, thus creating a vessel of which one side is formed by the filter cake itself. The shield is kept nearly filled with wash liquor in hydrostatic equilibrium with the prefilter pulp, so that the cake is supplied with wash liquor over the whole surface without possibility of areas being missed as by the usual sprays.

B. M. VENABLES.

Concentration and evaporation of liquids. SALT UNION, LTD., D. V. PLUMBRIDGE, and W. E. GIBBS (B.P. 289,116, 23.12.26).—The liquid (*e.g.*, brine) to be evaporated is heated and, in the form of a spray or film, is passed countercurrent to relatively cool air, which is heated by the brine and subsequently cooled to condense its moisture and re-used. The crystals from the brine are recovered by a conveyor or collected in a box and the excess brine is returned with new brine to the heater of the same evaporator, even when several effects are used in series, but in multiple effects the condenser of one effect forms the heater of the next.

B. M. VENABLES.

Concentrators, dehydrators, and like apparatus. [Evaporation by submerged flame.] C. F. HAMMOND and W. SHACKLETON (B.P. 289,159, 22.1.27).—The apparatus comprises a nest of stoneware burner tubes and inner and outer pots, so arranged that the gases of combustion form an air-lift circulator, and the concentrated solution is kept separate from the weak. Several evaporators may be arranged in series with a float-controlled inlet on the first and a regulatable draw-off valve on the last.

B. M. VENABLES.

Evaporators. J. HOLMES and H. A. KINGCOME (B.P. 289,717, 1.11.27).—An evaporator of the type comprising a (usually) cylindrical casing containing a nest of tubes heated externally by steam, with a large downcomer to one side of the heating tubes, is erected in an inclined position with the downcomer on the lowest side, and the lower end is suitably shaped for the ready discharge of crystals.

B. M. VENABLES.

Distillation apparatus. J. SCHNEIBLE (B. S. INGRAM, adtrix.), Assr. to SCHNEIBLE TRUST (U.S.P. 1,670,743, 22.5.28. Appl., 12.11.23).—The heating, distilling, and condensing sections of the still are arranged in the form of a vertical column, and the liquid to be distilled is introduced at an intermediate point in the distilling section. Somewhat above this point is a device, controlled by the temperature, which regulates the supplies of cooling and heating media to the condensing and heating sections, respectively.

F. G. CLARKE.

Separation of liquids of different specific gravities. W. A. WHITE, and WHITE OIL SEPARATORS, LTD. (B.P. 285,707, 7.6.27).—The mixed liquids (*e.g.*, oil and water) are caused by baffles to flow up and down several times within a tank, oil being drawn off at each upward flow. The baffles are so inclined that during each flow the passage converges in the direction of motion.

B. M. VENABLES.

Separation of liquids. O. L. BARNEBEY, Assr. to AMER. SOLVENT RECOVERY CORP. (U.S.P. 1,661,403, 6.3.28. Appl., 9.9.20).—Liquid mixtures are separated into their constituents by adsorption in a solid adsorption material, removal of unadsorbed diluent gases, and fractional evaporation.

C. O. HARVEY.

Centrifugal apparatus for the separation of suspended particles in a liquid. K. T. R. LUNDGREN (B.P. 282,681, 20.12.27. Swed., 22.12.26).—The separation is effected in tubes with closed ends supported on a link work so that their angle to the axis may be varied. The tubes may also be of flattened cross-section with the long axis radial or tangential as desired.

B. M. VENABLES.

Centrifugal separators [for the solid constituents of pulp]. F. M. COX (B.P. 289,681, 3.8.27).—The pulp is admitted to a centrifugal bowl rotating about a vertical shaft, and subjected to horizontal pulsations either by means of a piston-like baffle plate which is reciprocated vertically within the bowl or by pressure-water admitted intermittently, under control of a rotary valve, to a space between the wall of the bowl and an inner perforated wall. The heavier portions are discharged through ports which may be controlled by valves, and the lighter over the inner rim of the bowl.

B. M. VENABLES.

Machines for expressing liquids from solids. H. J. L. DUNLOP and A. J. SIBBALD (B.P. 289,954, 4.2.27).—The material is pressed by being caused to travel through an annular space, tapering in longitudinal cross-section, between a rotating member and a fixed member, the liquid emerging through channels left in the fixed outer member. The pressing action is obtained from another member rotating on an axis inclined to that of the machine in general, and at the same speed as the first-mentioned rotating member, giving a wobbling action.

B. M. VENABLES.

Apparatus for spraying and treating liquids. C. O. LAVETT, Assr. to BUFFALO FOUNDRY & MACHINE CO. (U.S.P. 1,667,291, 24.4.28. Appl., 11.8.24).—A horizontal cylinder, having shallow circumferential ribs, rotates in an axially disposed trough containing the liquid. In order to confine the adhering film of liquid in contact with the cylinder, so that the latter imparts its velocity to the liquid, a concentric, adjustable baffle is placed close to the rising surface of the cylinder. Above the baffle is a horizontal blade, which, by radial adjustment, enables the thickness of the film to be controlled; the surplus liquid falls into an adjacent receptacle which supplies the trough. The spray produced above the horizontal blade may enter a drying chamber, through which a current of hot air is passed, or it may impinge upon an internally-heated rotating

cylinder. By causing the spray to impinge upon a cooled surface, a liquid may be crystallised.

F. G. CLARKE.

Apparatus for cooling of fluids. O. SIMMEN (B.P. 282,717, 3.11.27. Switz., 28.12.26).—A casing contains a number of pipes of different diameter, the cooling agent being introduced through nozzles within the larger pipes and continuing through the smaller pipes, whilst the fluid to be cooled passes over the outside of the pipes and is guided by partitions so as to be subjected to a number of changes of direction.

B. M. VENABLES.

Treatment of gases. O. L. BARNEBEY, ASST. to AMER. SOLVENT RECOVERY CORP. (U.S.P. 1,661,104 and 1,661,149, 28.2.28. Appl., 21.1.25).—(A) In a tower containing activated carbon and provided with gas inlet and outlet, the temperature of the carbon is controlled by a pipe, in close proximity thereto, having separate connexions to heating and cooling supplies. (B) A mixture of gases to be separated is passed into activated carbon which retains the adsorbable constituents. The latter are subsequently expelled by applying heat indirectly to the interior of the carbon, and are cooled and collected.

H. HOLMES.

Dust catcher [for gases]. F. R. MCGEE (U.S.P. 1,659,607, 21.2.28. Appl., 18.12.25).—A main casing is provided near one end with an inlet for gas to be purified, and on opposite sides of the casing are arranged a conduit for distributing the gas and a main conduit for collecting the purified gas. Partitions prevent direct access from the inlet to the distributing conduit, and define first and second expansion chambers connected by a constricted passage in which is mounted a concave nozzle adapted to cause a sudden change in the direction of flow. The second expansion chamber is of a size to decrease the rate of flow considerably. Other separating chambers are provided, each communicating independently with the distributing conduit.

H. HOLMES.

Air filter. G. HAIN (U.S.P. 1,670,348, 22.5.28. Appl., 23.4.25).—The inlet and outlet have expanded inner ends, but the former has four radial ribs extending nearly to the centre. A screen to retain the filtering material is releasably secured over the outlet at its inner expanded end. An opening at the top of the filter has a cover and is large enough to permit removal of the screen.

F. G. CLARKE.

Air cleaner. J. HAASE and O. HIBNER, ASSTS. to J. HAASE (U.S.P. 1,670,660, 22.5.28. Appl., 27.6.27).—A receptacle containing water has a horizontal, submerged, perforated partition, below which constitutes a sediment chamber. The air from a vertical supply pipe, supported by the detachable lid of the receptacle, strikes a baffle-plate below the submerged end of the supply pipe and bubbles through the water. The upper portion of the cleaner contains fibrous packing upon a reticulated support and an anti-splash screen is placed at the water level.

F. G. CLARKE.

Recovery of absorbable substances from gaseous mixtures. E. C. R. MARKS. From CARBIDE & CARBON CHEMICALS CORP. (B.P. 291,277, 22.10.27).—Air charged with alcohol vapour, ether vapour, etc. is passed

upwards through a horizontal bed of active charcoal supported in a horizontal cylinder in such a manner as to leave spaces above and below it. When absorption is complete, the residual air in the spaces is expelled by evacuation or by a current of steam and passed together with fresh supplies of the charged air through a second absorber working alternately with the first. As soon as the air is expelled, steam is passed through the charcoal to expel the liquid condensed therein, and the mixed vapours pass to rectifying and condensing apparatus.

L. A. COLES.

Gas-analysis apparatus. ELECTROFLO METERS CO., LTD. From REPUBLIC FLOW METERS CO. (B.P. 288,510, 5.12.27).—An apparatus for the determination of a single constituent by absorption, in which the measuring burette is filled by a hand-operated, aspirator rubber bulb, and the contents of the burette are transferred to the absorber and back again by another water-filled rubber bulb.

B. M. VENABLES.

Anti-friction facing material for bearings etc. SOC. ANON. FRANÇ DU FERODO (B.P. 275,648, 5.8.27. Fr., 7.8.26).—A mixture of synthetic resin and fibrous filling materials is claimed; metal and graphite are excluded as fillers.

F. G. CROSSE.

Centrifuge. L. ALTPETER (U.S.P. 1,661,782, 6.2.38. Appl., 26.1.27. Ger., 8.7.25).—See B.P. 284,831; B., 1928, 320.

Treatment of steam to reduce or prevent corrosion. R. W. BAILEY, ASST. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,666,523, 17.4.28. Appl., 17.12.21. U.K., 20.12.20).—See B.P. 177,234; B., 1922, 358 A.

Fluid filters [for lubricants]. AC SPARK PLUG CO., ASSECS. of C. W. MCKINLEY (B.P. 267,485, 21.2.27. U.S., 11.3.26).

Extraction of extraneous material from fluids or gases. [Steam separator.] J. GORDON & CO., LTD. From HAGAN CORP. (B.P. 289,192, 9.2.27).

Distillation of liquids (B.P. 276,302).—See II. **Alloys for pistons** (B.P. 275,985).—See X. **Precipitation of particles from gases** (B.P. 290,030). **Pyrometers** (B.P. 290,493).—See XI. **Dehydration of liquids** (U.S.P. 1,670,101).—See XVII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Heat of coking of gas coals and coking coals. II. E. TERRES and M. MEIER (Gas- u. Wasserfach, 1928, 71, 457—461, 490—495, 519—523; cf. B., 1927, 177).—The method of calculating the heat of coking described previously (*loc. cit.*) needs correction; the heat given up by the volatile products should not be included. The results have been recalculated, making use also of the more accurate recent determinations of the specific heats of coke (B., 1928, 392). The new values are slightly lower, but, in general, the form of the curves showing the variation of the heat of coking with temperature is unchanged. The method of calibrating the apparatus is discussed; in determining the heat content of the empty system at 600° graphite is preferable to quartz as filling material. The heats of coking of

another nine gas coals, including three English coals (Easington, Ravensworth-Pelaw, and Boldon), have been determined for temperatures of carbonisation from 650° to 1050°. At the same time measurements have been made of the yield, composition, and calorific value of the gas evolved. Each coal appears to possess its own specific heat of coking-temperature curve, and it is impossible at present to formulate any general rules relating the heat of coking to coal composition or to temperature.

A. B. MANNING.

Replaceable bases in roofs of lignite seams. E. McK. TAYLOR (Fuel, 1928, 7, 227—228; cf. B., 1928, 288).—The p_H values of clays forming the roofs of lignite seams are generally lower than those of the roofs of bituminous coal seams, the main replaceable base present being calcium. Bacterial decomposition under a roof containing calcium aluminosilicic complex is limited by the acidity of the medium and the aerobic conditions; such decomposition results in the formation of lignite. If lignite is to be regarded as a stage in the formation of coal, it seems probable that, before the lignite can be converted into bituminous coal, base exchange between the roof and a solution of sodium chloride must take place, followed by hydrolysis of the product in fresh water, in order to provide a suitable medium and anaerobic conditions for the bacterial action.

A. B. MANNING.

Replaceable bases in roofs of anthracite seams. E. McK. TAYLOR (Fuel, 1928, 7, 228—229; cf. preceding abstract).—The roofs of anthracite seams, like those of bituminous coal seams, are alkaline and contain sodium as the main replaceable base. The conditions controlling bacterial activity during the formation of anthracite and bituminous coal respectively are therefore similar, and it is concluded that the differences in the type of coal formed must have their origin in differences in the starting material.

A. B. MANNING.

Base exchange and the formation of coal. E. McK. TAYLOR (Fuel, 1928, 7, 230—238; cf. preceding abstracts).—A theory of the formation of coal, based on investigations of the roofs of a large number of seams, aspects of which have already been discussed, is developed in more detail. Plant debris may undergo three different types of decomposition: (1) under acid conditions without a roof, leading to peat formation; (2) under acid conditions under a roof containing calcium aluminosilicic complex, leading to lignite formation; (3) under alkaline anaerobic conditions under a roof containing sodium aluminosilicic complex, leading to the formation of the members of the bituminous coal-anthracite series. The length of the peat stage preceding the third type of decomposition determines the type of coal ultimately formed. The theory is discussed in relation to the South Wales coal-field.

A. B. MANNING.

Microstructure of New Zealand lignites. W. P. EVANS (New Zealand J. Sci. Tech., 1928, 9, 339—359).—A further account of the Broken River lignites (see B., 1927, 690).

Use of graphical methods in the control of a coke-oven plant. G. W. J. BRADLEY (Fuel, 128, 7, 258—267).—The value of the use of graphical methods

is shown by their application to (a) the control of a crude benzol plant, (b) cost estimation, and (c) the calibration of storage tanks for stocktaking.

A. B. MANNING.

Determination of volatile matter in coke. F. J. EATON and S. PEXTON (J.C.S., 1928, 1215—1217).—Dry, finely-ground coke (1 g.), contained in a platinum crucible, is heated at 950° for 7 min. Oxidation losses are avoided by heating in an atmosphere of nitrogen. The results are consistent to within $\pm 0.05\%$.

F. J. WILKINS.

Isolation and detection of cellulose in peat. K. HESS and W. KOMAREVSKY (Z. angew. Chem., 1928, 41, 541—542).—A method for the isolation of cellulose from peat has been developed and applied to a sample of peat from the environs of Moscow. The air-dry material (8—10% of moisture) is extracted with ether (loss, 6%) and then with an alcohol-benzene (1:2) mixture (loss [bitumen], 10%). The extracted material is next shaken with 1% sodium hydroxide solution until no more coloured substances are removed (loss, 49%), and then subjected several times to the alternate action of a dilute (0.3—1%) solution of chlorine dioxide and 2% sodium sulphite solution (loss, 23%). The white, fibrous residue is dissolved in cuprammonium hydroxide and cellulose precipitated, after addition of alcohol, with acetic acid. The cellulose is washed with dilute acetic acid, water, alcohol, and, finally, ether. The purity of the sample is determined by measuring the rotatory power in cuprammonium hydroxide solution, or the rotatory power of the acetate in chloroform or pyridine-acetone solution. The present sample contained 10% of cellulose, calculated on the air-dry material. Whenever possible the purity of the cellulose obtained should be confirmed. The failure of Odén and Lindberg (B., 1926, 568) and Marcusson (B., 1927, 129) to do this renders their results open to criticism.

J. S. CARTER.

Formation of condensate in the transmission of gas under high pressures. K. BUNTE and A. KAMMÜLLER (Gas- u. Wasserfach, 1928, 71, 25—28).—Theoretical considerations governing the formation of condensate are discussed, but it is pointed out that compressor oil or tar acts similarly to benzol wash-oil and is capable of removing hydrocarbons from gas at partial pressures below saturation. The volume of a vapour such as naphthalene corresponding to saturation at any temperature is proportional to the ratio of the partial pressure to the total pressure. Hence with high compression the weight per unit volume of a vapour which the gas will carry is decreased proportionally. The average composition of town gas is discussed, and it is concluded that condensation of liquid hydrocarbons is unlikely at pressures below 35 atm. (This refers to gas which has been scrubbed for benzol.) Condensation of water and naphthalene is to be expected, and owing to the variable composition of town gas no general rule can be given. The conditions with oil gas are quite different. Whilst the use of 30 atm. is now proposed, pressures at present used in Germany do not exceed 4.5 atm. General experience in these cases shows for the most part that only a watery condensate is formed.

C. IRWIN.

Determination of the unburnt constituents of flue gases by combustion over copper oxide. H. MIES (Gas- u. Wasserfach, 1928, 71, 509—515).—The influence of temperature, time of contact, and other factors on the rate of combustion over copper oxide of a methane-nitrogen mixture containing 2% of methane has been studied. With a tube 18 mm. in diam. the temperature required for complete combustion with one passage of the gas varied from 770° to 900°, depending on the length of the copper oxide layer (40—10 cm.) and the gas velocity (2.5—10 litres/hr.). The presence of hydrogen, carbon monoxide, water vapour, or carbon dioxide does not affect the rate of combustion of the methane at these temperatures, but at lower temperatures the presence of hydrogen has a retarding influence. The rate of combustion is greatly influenced by the condition of the copper surface, increasing with successive reductions and oxidations. Before each series of measurements, therefore, the oxide was alternately reduced and oxidised until no further increase was observed. The results, however, indicate that there still exists a variation in the activity of the oxide along the tube. A. B. MANNING.

Gas-works effluent. W. P. SMITH (Gas J., 1928, 181, 690—692).—A review of the present situation concerning disposal of gas-works effluents shows that the difficulty is chiefly due to the presence of phenolic substances and certain salts which are found in the waste from ammonium sulphate plants. Reduction of phenolic contamination follows separation of the tar from the gas before condensation of the liquor, and, conversely, prolonged contact of tar and liquor will increase the phenolic content of the latter. Other methods of plant operation and treatment of condensates are described which will reduce the toxicity of the effluent. R. H. GRIFFITH.

Partial dehydrogenation process for certain hydrocarbons which favours the binding of the carbon. A. LÉAUTÉ and G. DUPONT (Compt. rend., 1928, 186, 1558—1560; cf. B., 1927, 737).—Fuel oil, coal tar, and similar mineral products, when heated at 250—350° in the presence of sulphur (2—10.5%) for a short time, lose almost all the admixed sulphur as hydrogen sulphide and yield highly viscous substances. G. A. C. GOUGH.

Behaviour of Emba crude oil in the refinery. E. PYNÄLÄ (J. Inst. Petroleum Tech., 1928, 14, 330—350).—Crude oils from the Emba region have d_{15}^{20} 0.845—0.904, and contain gasoline 0.26—7%, and kerosene 1.8—27.5%. The mazouts, like the original crude oils, are practically free from paraffin, and have, in consequence, very low setting points (—20°). Nevertheless, special precautions must be taken in distilling the mazouts if lubricating oils of low setting point are to be obtained. An Emba mazout having d_{15}^{20} 0.8843, flash point (Brenken) 136°, (Pensky-Martens) 113°, viscosity at 50° 2.4° (Engler), 0.165% of paraffin (Zaloziecki) of m.p. 56°, and 10% of asphalt (Akzise), was distilled (1) in a Nobel battery, (2) in the laboratory. The residues were 11.7 and 12.1% respectively; thus practically all the non-asphaltic constituents were removed in each of the distillations, which were there-

fore comparable. A kerosene fraction (13.8%) was produced in (1) but not in (2). Machine oil and cylinder oil fractions from (1) had lower sp. gr., lower viscosities, much higher setting points, and lower flash points; moreover, there was great difference in the flash points of these fractions from (1) as determined by the methods of Brenken and of Pensky-Martens. Apparently overheating had occurred during the large-scale distillation. This was confirmed by treating the machine oil and cylinder oil from the large-scale distillation with superheated steam for 2 hrs., whereby highly unsaturated impurities of low viscosity and low flash point were removed, leaving oils which compared favourably with the corresponding fractions produced on the small-scale distillation; moreover, the flash points of the purified oils now agreed. After being refined with 3.75% of 94% sulphuric acid, the machine oil distillate, although quite dry, was turbid at room temperature. Filtration gave a vaseline-like substance, which, after being washed with amyl alcohol and ethyl alcohol, was identified as ceresine, m.p. 72.5°. Ozokerite was found to be fairly volatile in superheated steam at 200°, and to be converted into ceresine thereby. Nevertheless it is impossible that the ceresine in the machine oil could be derived from the ozokerite; it is probable that ceresine was formed during distillation of the mazout, possibly from asphaltic-like and resinous constituents which the Emba crude oil also contains. Machine oils free from asphalt have been produced from Emba crude oils by treatment with sulphuric acid (e.g., 5% of acid containing 20% SO₃), and concentration by means of steam at 180—200°; the viscosity at 0°, but not at 50°, is thereby considerably raised. Acid-refined machine oils from Emba crude oil, although good in colour and other physical properties, show great tendency to emulsify, and cannot, therefore, be used in turbines. Such emulsifying agents may be removed by agitating the oil with an aqueous solution of the alkali soap derived from kerosene-naphthenic acids. In order that this treatment may not affect the flash point the naphthenic acids must first be freed from kerosene by steam-blowing the kerosene lye. W. S. NORRIS.

Recovery of gasoline from field and refinery gases with special reference to the Bayer charcoal process. I. EDELEANU (J. Inst. Petroleum Tech., 1928, 14, 296—313).—Further developments of natural gas production in Rumania are indicated. In a transportable plant having a daily production of 7 metric tons and weighing (building etc. included) only 60 tons, the gas is filtered and introduced at the bottom of one of several absorption vessels, containing the charcoal. These vessels are arranged in parallel; operations on them are scheduled so that adsorption periods in different adsorbers are evenly overlapping, and rinsing periods occur at equal time intervals and do not coincide in different adsorbers. If the gas is rich the heat generated may necessitate the circulation of cold water through internal coils to keep the temperature below 50°. The charcoal exhibits selective adsorption. Heavier gasoline fractions displace adsorbed lighter fractions; the lighter constituents of the portion of gasoline first adsorbed are gradually displaced upwards. The whole charge thus becomes saturated first towards

methane, then towards ethane, propane, etc. Gasoline fractions being mutually soluble, the fractionation thus obtained is not very sharp, but it is sufficiently so to be of practical use in determining the "end-point" of the gasoline which finally will be recovered. When the desired charge is reached, closed steam at about $2\frac{1}{2}$ atm. and 127° is used for steaming out. Later open steam is introduced at the top of the adsorber; the steam-vapour exit mixture is consequently rich in gasoline vapour until the end of the "rinsing" process. Economy in open steam is attained by passing the effluent vapours, on their way to the condenser, through the coils of a heat-interchanger or "evaporator" charged with water, which is thereby heated to 80 – 85° . The dome of the "evaporator" is connected with a steam ejector operated by the open steam used in rinsing. The resulting reduction of pressure in the evaporator to about 0.55 atm. causes the water therein to boil. Nearly half the steam for rinsing is regenerated, condensing equipment and cooling water consumption are diminished, and the feed of rinsing steam is, moreover, automatically regulated. If very rich gas is being treated, e.g., well trapped, uncondensed, still vapours, fresh gas is turned directly on to the wet charcoal, evaporation of the retained water then assisting the regulation of the temperature of reaction. Otherwise the continued use of closed steam and blowing with air serve to dry the adsorbent. During the entire process the charcoal is never heated above 140° , yet the extracted gasoline may have an "end-point" as high as 160° . This is largely due to the fact that the open steam furnishes an atmosphere in which the partial pressure of the gasoline is practically zero. The initial b.p. of the recovered gasoline may vary between 25° and 33° ; volume of distillate at 40° between 3% and 46%; and final b.p. between 103° and 163° . Overall steam requirements of the plant are 3 – $4\frac{1}{2}$ tons per ton of recovered gasoline, or 2 – $3\frac{1}{2}$ tons per ton in the actual process. The make-up of charcoal does not exceed 1 kg./ton of gasoline. A high sulphur content of the gas necessitates a previous scrubbing with lower-grade charcoal.

W. S. NORRIS.

Flame characteristics of "pink" and "non-pink" fuels. G. B. MAXWELL and R. V. WHEELER (J. Inst. Petroleum Tech., 1928, 14, 175–182).—Photographs have been obtained of the explosions of gaseous air-pentane and air-benzene mixtures, ignited by means of a sparking plug placed centrally in one end of a steel cylinder of variable length, fitted with a longitudinal glass window and with a Bourdon pressure gauge. Time-calibration of the photographs was obtained by recording, simultaneously with the explosion, a series of sparks from an electrically maintained mercury-break tuning fork. Pentane-air mixtures contained 2.8–3.8% of pentane by vol.; benzene-air mixtures about 3–3.5% of benzene. Initial pressures were from 1 to 2 atm. The following differences were revealed between an (audible) pink and an (inaudible) non-pink explosion. In the former, such as that of a pentane-air mixture at, say, 2 atm. initial pressure, a delay occurs in the process of combustion initiated in or immediately behind the flame front. Some additional shock or impulse is, apparently, required to complete

the process, and this is provided by the sudden arrestment of the accelerating, vibrating flame front at the end of the cylinder; the shock wave set up causes an almost instantaneous completion of the combustion throughout the cylinder, with a consequent very rapid increase in pressure. In a non-pink explosion (e.g., that of an air-benzene mixture) there is no delay in the combustion process, which is continuous and protracted, behind the wave front. Pinking, in fact, is due not to pre-ignition, but to very rapid, delayed after-burning. The longer the cylinder, the more readily was a pinking explosion produced. Turbulence, maintained by means of a fan, reduced the tendency to pink. Addition of lead tetraethyl vapour caused violent pinking, but the tendency to pink was eliminated by causing thermal dissociation of the lead tetraethyl, with formation of a smoke of metallic lead, previous to ignition of the mixture.

W. S. NORRIS.

Action of light on transformer oils. G. INCZE (Petroleum, 1928, 24, 743–744).—Increases have been observed in the tar numbers, tar-formation numbers, tar-formation numbers in presence of copper, and acid numbers of American, Russian, and Galician transformer oils which had been exposed to sunlight for periods up to 3 years, but no appreciable change was detected in samples of the same oils which had been preserved in the dark in the absence of air.

W. S. NORRIS.

"Gas benzine" of the Gelsenkirchener Bergwerks-A.-G. tar works. F. KROLPPFEIFFER and H. SEEBAUM (J. pr. Chem., 1928, [ii], 119, 131–156).—The "gas benzine," the volatile fraction condensed after the separation of the tar, after treatment with alkali, yielded the following fractions on repeated distillation: (1) b.p. 33 – 36° (2.9%), containing a pentane C_5H_{12} and Δ^2 -pentene, (2) 36 – 40° (1.5%), containing *n*-pentane and trimethylethylene, (3) 63 – 67° (4.0%), containing β -methylpentane and hexene, (4) 67 – 70° (4.2%), containing *n*-hexane and β -methyl- Δ^2 -pentene (the latter was synthesised by the elimination of water from dimethyl-*n*-propylcarbinol and from ethylisopropylcarbinol), (5) 79 – 82° (1.6%), consisting of a mixture of hexane and heptane (?) and benzene. In addition, the presence of cyclopentadiene (identified as dimethylfulvene) in fraction (2), of carbon disulphide in the fraction of b.p. 40 – 50° , of methyl ethyl ketone, and of minute amounts of acetone in practically all the fractions, was demonstrated. The physical characteristics of the isolated hydrocarbons are recorded.

I. VOGEL.

Development problems in exploitation of natural gas. S. J. M. AULD (J. Inst. Petroleum Tech., 1928, 14, 190–214).—For greater control the provision of high-pressure gas separators between the oil-collecting lines and the final low-pressure separators is desirable. The measurement of gas volume by observations of pressure drop in lines is discussed. Low-pressure gas may be measured by means of an orifice meter. Highly sulphurous gas, e.g., that of Masjid-i-Suleiman, containing up to 12% by vol. of hydrogen sulphide, can be used as fuel for boiler firing without preliminary desulphurising. Corrosion is only to be feared if there is direct impingement of incompletely burnt gas (which may contain elemental sulphur) on corrodible metal

parts, or if a cool metallic surface is present on which momentary condensation may occur of sulphurous or sulphuric acid. The presence of hydrogen sulphide renders the use of solid adsorbents impossible, either for the main extraction of condensable hydrocarbons from natural gas or for the final stripping; traces of air admitted with the gas, or with the steam during the regenerating process, cause deposition of sulphur in the pores of the adsorbent. The oil-absorption process for extracting gasoline from natural gas is discussed.

W. S. NORRIS.

Cracking of hexadecane in presence of catalysts.

H. GAULT and R. SIGWALT (Ann. office nat. comb. liq., 1927, 2, 309—323, 543—584).—Comparable results are obtained only when identical volumes of hexadecane are employed; the light products eventually undergo polymerisation and resinification. The cracking reactions take place irregularly, centres of dissociation being formed. Aromatic hydrocarbon are produced from light ethylenic hydrocarbons, and not from acetylene. In the absence of chemical catalysts, fracture of the chain occurs indifferently at any point, the point tending to approach the end of the chain with rise of temperature; the resulting double linking is preferably located in the shorter chain. The composition of the gases varies within narrow limits for particular temperatures; differences in the composition of the final products are mainly due to secondary reactions. Deposits of carbon on pumice reduce the activity of the catalyst at 400° and 450° without exerting any specific action; at higher temperatures the effect is less noticeable. The gases obtained by cracking hexadecane in presence of nickel consist almost exclusively of methane and hydrogen; the liquid products consist of aromatic and saturated acyclic hydrocarbons, ethylene hydrocarbons being found only at 350°. The activity of reduced nickel at first increases and then diminishes. Cracking of hexadecane in presence of anhydrous aluminium chloride yields butane with a little propane and heavier vapours, and an asphaltic residue of low hydrogen content; the hexadecane molecule yields approximately three butane molecules.

CHEMICAL ABSTRACTS.

Decomposition of paraffin wax on heating.

H. I. WATERMAN, J. N. J. PERQUIN, and H. A. VAN WESTEN (J. Inst. Petroleum Tech., 1928, 14, 318—327).—The mol. wt. of Rangoon paraffin found by the Rast method (B., 1922, 393 A) was 427 and 417. Residues from Engler distillations carried out during experiments on cracking and berginisation of wax (cf. Waterman and Perquin, B., 1927, 643; 1925, 746) showed little difference in mol. wt., which was, however, lower than that of the original wax. Rangoon wax of mol. wt. 410 has been heated in an atmosphere of dry nitrogen at 327—370° for periods up to 20 hrs. The wax is comparatively stable up to 370°. Heating for 20 hrs. at 360° or for 10 hrs. at 370° caused the elimination of an unsaturated distillate, leaving unsaturated residues of mol. wt. 369 and 364, respectively, and correspondingly lowered molecular refractions. This treatment caused a decided lowering of the setting point (Shukoff) of the residue (from 58° to about 54°), an effect which was brought about to a less pronounced degree even at 327° by prolonged heating (16 hrs.).

Extraction and refining of paraffin wax. W. ALLAN (J. Inst. Petroleum Tech., 1928, 14, 328).—Extraction of wax is usually accomplished by pressing a chilled, cracked distillate, which has preferably been redistilled rapidly, using a minimum of steam. The scale is then melted, sweated to the required m.p., and refined.

W. S. NORRIS.

Determination of paraffins in commercial benzole and motor petrols. R. HEILINGÖTTER (Chem.-Ztg., 1928, 52, 437—438).—Examination of hydrocarbon mixtures such as motor spirit depends chiefly on solubility of aromatic or paraffin constituents in various solvents, or their behaviour with sulphonating agents, methyl sulphate, etc.; the latter does not give quantitative results, nor is treatment with acetic anhydride satisfactory. Mixtures containing benzene can be tested by use of a refractometer, provided no substances such as acetone, alcohol, or tetralin are present; curves are given showing the relation between refractive index and the benzene content.

R. H. GRIFFITH.

Physico-chemical basis for the cementing of oil wells with Portland cement. I. A. ALEKSANDROV (Nef. Khoz., 1927, 13, 429—438).—Crude oil forms emulsions with cement; gum arabic prevents the formation of emulsions. The effect of various constituents of the oil or of substances produced by the oil-cement mixture on the formation and stability of the emulsions is considered.

CHEMICAL ABSTRACTS.

Action of coal on photographic plates. HASLAM.—See XXI. **Disinfectants.** TAYLOR.—See XXIII.

PATENTS.

Method of saving fuel. J. M. W. KITCHEN (U.S.P. 1,659,692, 21.2.28. Appl., 17.6.24).—The fuel is composed of a mixture of relatively large hard coal particles and a matrix of smaller particles of moistened coal relatively rich in volatile matter and occupying only a portion of the interstices between the larger particles.

C. O. HARVEY.

Treatment of flue dust. K. M. SIMPSON (U.S.P. 1,661,636, 6.3.28. Appl., 7.4.26).—Flue dust is mixed with a pulverised carbonaceous fuel and a hydrocarbon and briquetted. The mixed materials are heated first in air and then at a higher temperature in the absence of oxygen, whereby the bulk of the volatile matter is removed and the materials are bonded together.

A. B. MANNING.

Coke and gas-oven and process for heating the same. C. WESSEL (B.P. 284,606, 26.1.28. Ger., 31.1.27).—Coke ovens with vertical heating flues have a mixing chamber beneath each flue in which an initial volume of preheated air is supplied to the gas, for the purpose of dilution and partial combustion. From each mixing chamber a pipe provided with lateral openings at various heights extends into the heating flue, the gas as it issues from these openings being burnt with secondary air supplied to the lower end of the flue. If a weak gas, e.g., producer gas or blast-furnace gas, is used for heating, it is preheated by passage through a regenerator and passed directly through the mixing chambers to the flues without admixture with primary air.

A. B. MANNING.

Semi-portable carbonising oven for fuel balls made of sawdust or other wood waste. L. DUPONT (B.P. 282,058, 6.12.27. Fr., 8.12.26).—The oven comprises a furnace capable of heating simultaneously (a) a tunnel chamber open at both ends, in which the fuel balls of sawdust or other waste material are semi-carbonised, (b) retorts in which the carbonisation of the fuel balls can be completed, and (c) chambers maintained at a lower temperature for reheating the fuel balls after semi-carbonisation. The fuel passes successively through a dryer, a stirring apparatus, and a hopper to the moulds, which are arranged on a moving endless horizontal belt, and in which the fuel is conveyed through the tunnel chamber. The distillation gases are passed through a tar remover and are returned thence to the furnace. A. B. MANNING.

Composition of fuel briquettes. R. LESSING (B.P. 290,380, 21.2.27).—In the manufacture of briquettes containing a small proportion of a salt or oxide of a multivalent metal as a catalyst, a coal is used which has been separated by flotation, and the bath liquor is removed by displacement with a continuous body of liquid of which the last portion is a solution or suspension of the catalyst to be added. A. B. MANNING.

Apparatus for making carbon and generating steam. L. J. McNUTT (U.S.P. 1,670,686, 22.5.28. Appl., 10.2.26).—The flame from a gas burner impinges upon a revolving, horizontal cylinder, which constitutes the steam generator. The deposited carbon is removed from the outside of the cylinder, and is collected in a receiver placed below. The steam leaves the cylinder through its hollow axle, and passes into a discharge pipe supported by the bearings and extending along the top of the cylinder. A battery of cylinders may be arranged above a single carbon receiver.

F. G. CLARKE.

Operation of internal-combustion engines I. G. FARBENIND. A.-G. (B.P. 274,462, 11.7.27. Ger., 13.7.26).—Engines of the Diesel type are operated on a pulverulent mixture of mineral coal and lignite, containing up to 80% of the former. A. B. MANNING.

Manufacture of mixed water-gas and distillation gas from fuels of volatile content. HUMPHREYS & GLASGOW, LTD., Asses. of C. S. CHRISMAN (B.P. 279,059, 27.9.27. U.S., 13.10.26).—The plant comprises two generators, two superheaters, and two combined carburettors and regenerators. The fuel bed in each generator has a lower gasification zone and an upper carbonisation zone. In operation, the two generators are blasted in parallel, the blast traversing the gasification zones only, and the blast gases passing successively through the respective superheaters, in which they are burnt with secondary air, the carburettors and regenerators, to the stack. After the blasting operation a down-run with steam is made on one generator and an up-run with steam on the other. The water-gas from the former is carburetted, and then passes through the superheater to the second generator, traversing the carbonising zone of the latter in admixture with the water-gas from the lower zone of the same generator. Some alternative methods of operation involving the same basic idea are described. A. B. MANNING.

Generation of water-gas. L. B. MOORHOUSE (U.S.P. 1,660,202, 21.2.28. Appl., 19.7.20).—Streams of air and steam are alternately passed diagonally downwards through a mass of incandescent fuel, the direction of flow being varied so that the path of one stream of air crosses that of the next. C. O. HARVEY.

Catalytic gas reactions. I. G. FARBENIND. A.-G. (B.P. 274,904, 25.7.27. Ger., 24.7.26).—In catalytic gas reactions, especially those carried out under pressure, and in particular in the destructive hydrogenation of carbonaceous materials, the finely-divided or granular catalyst is brought into suspension uniformly throughout the reaction space by means of an eddying gas current. Any dust evolved from the catalyst is removed from the reaction gases, by means of baffles or a dust separator, before they reach the cooler parts of the apparatus. A. B. MANNING.

Cracking of hydrocarbons. Crude-oil preheater. W. L. GOMORY, Assr. to C. M. LEONARD (U.S.P. 1,670,804—5, 22.5.28. Appl., [A] 14.2.20, [B] 30.4.21).—(A) 80–85% of a mixture of hydrogen and hydrocarbons is vaporised by forcing it through a conduit at about 538° and under about 150 lb. (B) Crude oil to be preheated passes down a hollow column and chamber situated centrally in a heated, vertical, cylindrical tank, and is then discharged uniformly into the tank, near the top of which the outlet tube is situated. C. O. HARVEY.

Refluxing tower [for mineral oils]. R. CROSS, Assr. to GASOLINE PRODUCTS Co. (U.S.P. 1,670,762, 22.5.28. Appl., 29.9.24).—In a dephlegmating tower of the type wherein vapours are bubbled through pools of oil contained in trays, the vapours are used to preheat the oil stock, which may also be partially diverted to any or all of the trays. C. O. HARVEY.

Distillation of liquids, more particularly of liquid hydrocarbons. M. SINGER (B.P. 276,302, 19.4.27. Ger., 19.8.26).—The oil, preheated in a tubular still, is automatically fed, with or without steam or a gas, and intermittently in predetermined quantities at regular intervals, to an evaporating tower. A number of such towers may be used. C. O. HARVEY.

Treatment of oil. F. A. KORMANN, Assr. to UNITED REFINERIES Co. (U.S.P. 1,661,804, 6.3.28. Appl., 14.2.21).—Oils of high b.p. are converted into lower-boiling products by distilling in the presence of steam in a still containing a mass of highly-absorbent brickwork, into the pores of which steam is introduced. C. O. HARVEY.

Catalytic apparatus [for oils]. E. T. HESSLÉ and W. LELGEMANN (U.S.P. 1,661,827, 6.3.28. Appl., 26.4.26).—Oil in the form of vapour or "fog" is treated by passing it through a fluid catalyst. C. O. HARVEY.

Distillation of hydrocarbons. M. R. MEACHAM, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,661,189, 6.3.28. Appl., 25.6.23).—A still provided with perforated steam-inlet pipes has a vertical tubular circulator fitted with a steam jet, whereby the oil is forced upwards into a tray, whence it flows downwards again in a number of small streams. C. O. HARVEY.

Oxidation of hydrocarbon oils. E. E. REID, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,663,523,

20.3.28. Appl., 5.7.21).—A gas containing oxygen issues in fine streams from a member rotating at a speed of over 1000 r.p.m. and situated below the surface of the hot oil. C. O. HARVEY.

Production of acids [by oxidation of hydrocarbon oils]. E. E. REID and C. E. BURKE, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,663,524, 20.3.28. Appl., 14.2.25).—A gas containing oxygen is passed into the oil, which is heated at below 300° and rapidly stirred. An intimate mixture of oil and gas is produced by causing the streams of gas to impinge on the stirrer and the oil in close proximity thereto. C. O. HARVEY.

Manufacture of unsaturated hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 290,322, 10.1.27).—Unsaturated hydrocarbons, in particular acetylene, are produced from heavy oils by spraying the latter into a combustible gas, or admixed with such a gas, in such a way that they pass through a high-temperature zone formed by the combustion of part of the mixture with oxygen or gases rich in oxygen, the combustion being so effected as to produce a disc-shaped flame. A. B. MANNING.

Preservation of mineral oils. F. HOFMANN and M. DUNKEL (B.P. 289,909, 5.11.26).—The rate of oxidation of mineral oils such as transformer and lubricating oils is retarded and their liability to form sludgy deposits is lessened by the addition of about 0.1% of a cyclic amine or similar highly basic compound (*e.g.*, piperidine, diethylaniline, phenylenediamines). [Stat. ref.] C. O. HARVEY.

Retort for carbonising bituminous fuels. J. PLASSMANN (U.S.P. 1,671,448, 29.5.28. Appl., 10.7.25. Ger., 30.9.24).—See B.P. 240,800; B., 1926, 228.

Production of high-grade products from raw coal. CHEM. FABR. IN BILLWÄRDER VORM. HELL & STHAMER A.-G., and F. L. KÜHLWEIN (B.P. 286,456, 5.3.27).—See U.S.P. 1,660,009; B., 1928, 325.

Coke-extracting mechanism for vertical retorts for distillation of carbonaceous materials. F. J. and E. WEST (U.S.P. 1,672,442, 5.6.28. Appl., 6.6.27. U.K., 28.6.26).—See B.P. 272,691; B., 1927, 674.

Calcining of coke [for electrodes]. V. C. DOERSCHUK and F. C. FRARY, Assrs. to ALUMINUM Co. OF AMERICA (U.S.P. 1,671,673, 29.5.28. Appl., 22.4.26).—See B.P. 269,849; B., 1928, 6.

Fuel for use in internal-combustion engines etc. E. G. E. MEYER (U.S.P. 1,671,158, 29.5.28. Appl., 5.6.26. U.K., 12.6.25).—See B.P. 262,363; B., 1927, 134.

Production of filling for dissolved gas [storage of explosive gases]. J. POMMÉE (U.S.P. 1,672,688, 5.6.28. Appl., 8.5.25. Ger., 5.5.24).—See B.P. 241,468; B., 1926, 40.

Conversion of hydrocarbons. H. NIELSEN, Assr. to B. LAING (U.S.P. 1,672,081, 5.6.28. Appl., 7.3.25. U.K., 3.12.21).—See B.P. 198,385; B., 1923, 760 A.

Distillation and cracking of hydrocarbon oils. H. J. JANSEN (U.S.P. 1,672,459, 5.6.28. Appl., 13.7.25. U.K., 18.7.24).—See B.P. 278,235; B., 1927, 900.

Catalyst for manufacture of hydrocarbons. L. EDELEANU, Assr. to ALLGEM. GES. F. CHEM. IND.

(U.S.P. 1,671,517, 29.5.28. Appl., 23.2.26. Ger., 19.12.24).—See B.P. 244,697; B., 1926, 396.

Tanks for transportation of volatile liquids. E. BRAINICH (B.P. 291,321, 18.1.28. Ger., 30.6.27).

Gas-analysis apparatus (B.P. 288,510).—See I. Lead and tin tetra-alkyls (B.P. 290,444). **Hydrogen from hydrocarbons** (B.P. 291,244).—See VII. **Asphaltic mixture** (U.S.P. 1,671,070).—See IX. **Polishes and soaps** (B.P. 261,039 and 287,114).—See XII. **Oil varnishes** (B.P. 287,115—6).—See XIII.

III.—ORGANIC INTERMEDIATES.

Preparation of acetic acid and its derivatives. E. TERLINCK (Chem.-Ztg., 1928, 52, 249—250, 270—272, 307—309, 326—328).—Barium acetate can be readily prepared anhydrous by evaporation of its solution over an oil-bath. For the preparation of barium acetate from commercial "grey acetate" the latter is dissolved in 3—3.5 pts. of water, and the filtered solution is treated with the theoretical quantity of sulphuric acid to liberate all the acetic acid present. The calcium sulphate precipitate is removed and the solution treated with a small quantity of barium acetate solution to remove sulphuric acid, filtered, and neutralised with barium carbonate. The clear yellow liquid is evaporated to d 1.40; on cooling, coarse crystals of barium acetate separate out. They are separated from the slightly warm syrupy mother-liquor by centrifuging, washed with a saturated solution of the pure salt, heated at 260° to decompose any tarry matter, redissolved, and recrystallised by evaporation of the filtered solution. The crude mother-liquor from the first crop is evaporated, the dry residue heated at 260° and dissolved in water, and the solution decolorised with charcoal and added to that obtained from the crystals. Barium acetate may also be prepared directly from crude pyrolygneous acid by treating it with an excess of baryta to precipitate tarry matter, evaporating the filtrate to a syrup, again filtering or centrifuging, and proceeding as before. Sodium acetate of high purity is readily prepared from the barium salt by double decomposition with sodium carbonate, and the regenerated barium carbonate is available for the preparation of further quantities of barium acetate. Glacial acetic acid (m.p. 15.7—16.0°) may be obtained directly from the granular barium salt by passing a current of dry hydrogen chloride through a copper tube packed with the granules and redistilling the acid over a small quantity of barium acetate, or by treating a saturated solution of barium acetate in glacial acetic acid (510 g./litre) with dry hydrogen chloride or with anhydrous sulphuric acid. Acetic anhydride is prepared from barium acetate by passing carbonyl chloride through the granulated salt in a copper still or through a suspension of the finely-divided acetate in acetic anhydride. The recovery of barium carbonate from the chloride or sulphate produced in the processes described above may be effected as follows: barium sulphate is reduced at 800° in a stream of water-gas, and the solution of barium sulphide is precipitated with sodium carbonate to obtain barium carbonate and pure sodium sulphide; barium chloride is treated with ammonia and carbon dioxide obtained by calcining

magnesite, whereby barium carbonate is precipitated, the ammonium chloride produced is distilled with sulphuric acid to regenerate hydrogen chloride for use again in the process; and, finally, the ammonium sulphate is heated with magnesia to regenerate ammonia and form magnesium sulphate.

A. R. POWELL.

Determination of the cresols. II. K. K. JÄRVINEN (Z. anal. Chem., 1928, 73, 446—451; cf. B., 1927, 596).—To obtain a quantity of the pure cresols from lysol for standardising the bromide-bromate solution used in the method previously described, the following procedure is recommended: 50 g. of lysol are dissolved in 25 c.c. of 15*N*-sodium hydroxide and 100 c.c. of water, and the hot solution is precipitated with 20 c.c. of 3*N*-barium chloride solution, diluted to 250 c.c., and filtered. The filtrate is treated with 35 c.c. of hydrochloric acid (*d* 1.18) and 40 g. of sodium chloride in a separating funnel. The cresols which separate are collected and distilled, the fraction coming over above 180° being collected, heated to boiling for a few minutes to expel moisture, and used immediately for making a standard solution of 10 g./litre. For the determination of cresols in relatively pure preparations, 10 g. are dissolved in 7 c.c. of 15*N*-sodium hydroxide. The solution is diluted to 100 c.c. and filtered, 10 c.c. of the filtrate are transferred to a butyrometer tube and treated with 11 c.c. of 1:1 hydrochloric acid previously saturated with cresol and 2 g. of sodium chloride, the mixture is shaken vigorously, heated at 50° for 15 min., and centrifuged, and the volume of separated cresol read off; 7.68 butyrometer degrees are equivalent to 1 g. of cresol.

A. R. POWELL.

PATENTS.

Manufacture of acetic anhydride. CONSORT. F. ELEKTROCHEM. IND., G.M.B.H. (B.P. 272,951, 17.6.27. Ger., 17.6.26).—Prior to contact with a catalyst (*e.g.*, molten alkali phosphate) giving rise to the formation of anhydride, acetic acid vapour is preheated at 400—800° in a chamber lined with material such as chromium or nickel alloys, silicon carbide, carbon, or graphite. The output of anhydride is greatly increased. Preheating may be external by means of hot gases, or internal by means of electrically heated resistances of carbon, graphite, chromium or nickel alloys, etc. B. FULLMAN.

Manufacture of the anhydrides of acetic acid, its homologues, and halogen derivatives. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 289,959, 4.2.27).—Sodium acetate (328 pts.), acetic acid (100 pts.), and silicon tetrachloride (170 pts.) are mixed in a closed vessel, and when the pressure has become normal again acetic anhydride and acetic acid are distilled off, the former in 98% yield. The ratio of sodium acetate to silicon tetrachloride (part of which may be replaced by titanium or tin tetrachlorides) must be substantially as given.

C. HOLLINS.

Manufacture of anhydrides of aliphatic acids. H. DREYFUS (B.P. 289,972, 8.2.27).—Acetic (or other aliphatic) acid of any strength is passed through a copper or fireclay tube at 800—1000° and then over sodium pyrosulphate, sodium hydrogen sulphate, phosphoric acid, or other suitable dehydrating agent at 150—200°.

C. HOLLINS.

Production of aldehydes from dicarboxylic acids. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 290,319, 7.1.27).—The acid or its anhydride is passed with a reducing gas (hydrogen, carbon monoxide) over a catalyst (iron, chromium, copper, manganese, cobalt, or their oxides) at 250—400°. Phthalic anhydride gives benzaldehyde, succinic anhydride propionaldehyde. Maleic anhydride may be converted first into succinic anhydride by hydrogenation at 180°.

C. HOLLINS.

Manufacture of crotonaldehyde. ELEKTRIZITÄTSWERK LONZA, Assees. of E. LUSCHER (B.P. 270,764, 9.5.27. Switz., 8.5.26).—Acetaldehyde free from acid is treated with 0.02% of caustic alkali in the form of 5% solution in an atmosphere free from oxygen. After aldolisation at 30—40°, the alkali is neutralised with phosphoric acid (no precipitation of salt occurs), and the product distilled directly at about 130°. The yield of crotonaldehyde is 95—98%.

C. HOLLINS.

Aldehyde-amine condensation products. GRASSELLI CHEM. CO., Assees. of W. B. BURNETT and I. WILLIAMS (B.P. 265,931, 29.12.26. U.S., 12.2.26; cf. B.P. 265,930, B., 1928, 420).—An amine (aniline, *o*-toluidine, *n*-butylamine, methylamine, ethylenediamine, *o*-tolylidiguamide, ethylaniline, diethylamine, etc.) is condensed with 2 or more mols. of an aldehyde containing more than two carbon atoms, in presence of acids or acidic condensing agents (acetic, propionic, butyric, valeric, stearic, oleic, picric, salicylic, hydrochloric, sulphuric, and phosphoric acids, or their amine salts, or zinc chloride, acid anhydrides, acid halides, etc.), the amount of the latter being preferably about 0.2 mol. for each mol. of amine. The products from primary amines and aldehydes other than α -unsaturated aldehydes are vulcanisation accelerators distinguished from those of B.P. 263,853 (B., 1927, 757) in that they are unaffected by the presence of carbon black in the rubber mix. Examples are: aniline with 5 mols. of *n*-butaldehyde and 0.05—0.15 mol. of *n*-butyric acid, or 0.5 mol. of sulphuric acid, or 1 mol. of stearic acid, or 0.15 mol. of acetic acid; aniline with 3 mols. of *n*-butaldehyde and 0.15 mol. of *n*-butyric acid; *o*-toluidine with 5 mols. of *n*-butaldehyde and 0.15 mol. of *n*-butyric acid; *n*-butylamine with 3 mols. of heptaldehyde and 0.15 mol. of *n*-valeric acid. C. HOLLINS.

Manufacture of diazotised monoamines of the cyclic series. I. G. FARBENIND. A.-G. (B.P. 268,789, 30.3.27. Ger., 30.3.26).—Arylamines containing negative substituents which make diazotisation difficult are converted into sulphamic acids, which are readily diazotised (with expulsion of the sulphonic group) in aqueous solution or suspension. Examples are: 2:5-dichloroaniline, 2:3- and 2:5-dichloro-*p*-toluidines, 3:4:5-trichloroaniline, picramic acid, aminoazo-benzene and -toluene.

C. HOLLINS.

Manufacture of new depolymerisation products from carbohydrates of high mol. wt. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 290,377, 15.2.27).—Carbohydrates are heated in boiling glycol or ethylene chlorohydrin or mixtures of these until the desired degree of de-association is reached ($\frac{1}{4}$ to $2\frac{1}{2}$ hrs.), and

the solvent is removed by distillation in a vacuum. The chlorohydrin dissolves about 8% of cellulose.

C. HOLLINS.

Introduction of sulphocyanide [thiocyano-] groups into organic compounds. I. G. FARBENIND. A.-G., Assees. of H. P. KAUFMANN (B.P. 257,619, 26.8.26. Ger., 27.8.25).—Organic compounds are converted into thiocyanate-derivatives by treatment with a solution of a thiocyanate and a halogen. Ethylene and chlorine are led into a solution of sodium thiocyanate in 96% acetic acid to give dithiocyanoethylene, m.p. 90°, which is also formed by passing ethylene and bromine into a solution of sodium thiocyanate in 15% hydrochloric acid. $\alpha\beta$ -Dithiocyanophenylethane, m.p. 101°, is obtained from styrene, $\alpha\beta$ -dithiocyano- β -*p*-anisylpropane, m.p. 81°, from anethole, 4:4'-dithiocyanodiphenylamine, m.p. 120°, from diphenylamine, 5-thiocyanosalicylic acid, m.p. 165°, 4-thiocyano- α -naphthol, m.p. 113°, 2:4-dithiocyano- α -naphthol, m.p. 118—119°, 2:4-dithiocyano- α -naphthylamine, m.p. 204°, and 1-thiocyano- β -naphthol, m.p. 261° (decomp.), are similarly prepared. Antipyrine gives a 4-thiocyano-derivative, which may be hydrolysed in solution with dilute alkali to the 4-thiol compound, isolated as the 4:4'-disulphide, m.p. 256°.

C. HOLLINS.

Preparations suitable for production of aqueous solutions of organic compounds insoluble or difficultly soluble in water. I. G. FARBENIND. A.-G. (B.P. 290,554, 26.10.26. Addn. to B.P. 261,720; B., 1928, 455).—Mixtures of the sulphonic acids (or salts) mentioned in the prior patent with water-soluble salts, such as sodium sulphate, sodium chloride, etc., are used for solubilising organic liquids insoluble in water. An example is naphthalene, sulphonated and condensed with *sec*-butyl alcohol, and the reaction product neutralised with alkali and evaporated. [Stat. ref.]

C. HOLLINS.

Purification of aromatic hydrocarbons. H. G. C. FAIRWEATHER. From SELDEN Co. (B.P. 290,840, 7.6.27).—The crude aromatic oil, freed from bases and phenols, is given a partial wash with sulphuric acid, and is then treated with chlorine, in presence or absence of a carrier or catalyst, washed with water, and the purified hydrocarbons are distilled off. The chlorinated residue may be used as a solvent.

C. HOLLINS.

Preparation of H-acid [8-amino- α -naphthol-3:6-disulphonic acid]. I. GUBELMANN and J. M. TINKER, Assrs. to NEWPORT Co. (U.S.P. 1,670,406, 22.5.28. Appl., 4.11.25. Cf. U.S.P. 1,573,056; B., 1926, 528).— α -Naphthylamine-3:6:8-trisulphonic acid is fused with caustic alkali in the presence of added ammonia.

B. FULLMAN.

Manufacture of ketones of the anthracene series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 289,585, 16.2.27).—Anthracene is condensed with aliphatic acid halides in presence of aluminium chloride to give, according to conditions, 9-anthracyl alkyl ketones or a separable mixture of 1- and 2-anthracyl alkyl ketones, the latter predominating. The 1- and 2-anthracyl compounds are oxidised to the corresponding anthraquinonyl ketones: the *meso*-compounds yield anthraquinone on oxidation, or are isomerised to

1- and 2-compounds by treatment with aluminium chloride. *Anthracyl methyl ketones* (1-, m.p. 103—105°; 2-, m.p. 183—185°; 9-, m.p. 80°), 1- and 2-*anthraquinonyl methyl ketones*, m.p. 158—160° and 140—142°, respectively, 1- and 2-*anthracyl ethyl ketones*, m.p. 150° and 162—163°, respectively, and a *diacetylanthracyl quinone*, m.p. 212—215°, which is oxidised to a *diacetylanthracyl quinone*, m.p. 318—319°, are described; a second *diacetylanthracyl quinone* has m.p. 173°.

C. HOLLINS.

Manufacture of tetranitrodianthrone and of 2:7-dinitroanthraquinone therefrom. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 289,958, 4.2.27).—Dianthrone, nitrated at 0—5° with mixed acid, yields tetranitrodianthrone, the dienolic form of which (obtained by action of pyridine, aniline, dilute alcoholic alkali, etc. on the diketone) is oxidised with nitric acid or chromic-acetic acid to 2:7-dinitroanthraquinone, m.p. 284—285°.

C. HOLLINS.

Preparation of ethylidene diacetate. M. J. L. LEDRU and E. J. BACHMANN, Assrs. to Soc. CHIM. DES USINES DU RHÔNE (U.S.P. 1,672,646, 5.6.28. Appl., 22.1.26. Fr., 18.9.25).—See B.P. 252,640; B., 1926, 692.

Preparation of dithiazyl disulphide. C. M. CARSON, Assr. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,661,998, 6.3.28. Appl., 7.9.26).—See B.P. 277,338, B., 1928, 361.

Manufacture of 3:4-diaminobenzoyl-*o*-benzoic acids. NEWPORT Co. (B.P. 277,285, 10.1.27. U.S., 13.9.26).—See U.S.P. 1,663,229; B., 1928, 440.

Manufacture of alkyl derivatives of diphenolisatin. A. BLANKART, Assr. to HOFFMANN-LA ROCHE CHEM. WORKS (U.S.P. 1,667,239, 24.4.28. Appl., 7.7.27. Switz., 9.10.26).—See B.P. 278,672; B., 1928, 548.

Production of anthraquinonesulphonic acids. J. THOMAS, Assr. to SCOTTISH DYES, LTD. (U.S.P. 1,671,455, 29.5.28. Appl., 19.6.24. U.K., 31.8.23).—See B.P. 229,719; B., 1925, 349.

Catalyst carrier [for oxidation of organic compounds]. C. R. DOWNS (U.S.P. 1,672,308, 5.6.28. Appl., 13.11.19).—See B.P. 153,877; B., 1922, 197.

Unsaturated hydrocarbons (B.P. 290,322).—See II. **Catalysts** (B.P. 290,399).—See VII.

IV.—DYESTUFFS.

PATENTS.

Readily dispersible dye preparations. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, A. SHEPHERDSON, and A. J. HAILWOOD (B.P. 290,542, 22.7.27).—The products obtained by moderated oxidation of ligninsulphonic acid (cf. F.P. 374,162) possess in an enhanced degree the dispersing and solubilising properties of the starting-material, and are non-hygroscopic. When they are incorporated with a finely-divided dye paste, the latter may be dried at moderately high temperatures without losing its ready dispersibility in water.

C. HOLLINS.

Manufacture of water-soluble dinitroarylamino-diarylamines. I. G. FARBENIND. A.-G. (B.P. 279,133, 18.10.27. Ger., 18.10.26).—A *p*-aminodiphenylamine is

condensed with a 1-halogen-2:4- (or 2:6-)dinitrobenzene containing in position 6 (or 4) a carboxyl, carbalkoxyl, carbonarylalkylamide ($\cdot\text{CO}\cdot\text{NArR}$), sulpharylalkylamide ($\cdot\text{SO}_2\cdot\text{NArR}$), or sulphonic aryl ester group, to give yellow to reddish-brown dyes for wool, silk, and particularly for cellulose esters and ethers. From 4-amino-4'-methyldiphenylamine-2-sulphonic acid and 4-chloro-3:5-dinitrobenzenesulphonmethylanilide a brown wool dye is obtained. Other condensations are: 4-amino-4'-methyldiphenylamine-2-sulphonic acid with 4-chloro-3:5-dinitrobenzoic acid (yellow-brown on wool); *p*-aminodiphenylamine with 4-chloro-3:5-dinitrobenzoic acid (intense brown on acetate silk); 4-amino-4'-methoxydiphenylamine-2-carboxylic acid (prepared from 2-chloro-5-nitrobenzoic acid and *p*-anisidine) with 4-chloro-3:5-dinitrobenzoic acid; 4-amino-2-sulphodiphenylamine-2'-carboxylic acid with 4-chloro-3:5-dinitrobenzenesulphonmethylanilide; *p*-aminodiphenylamine with 2-chloro-3:5-dinitrobenzoic acid.

C. HOLLINS.

Manufacture of sulphur dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 289,241, 29.3.27).—Sulphur is introduced in the form of thiocyno-groups into intermediates for sulphide dyes by treatment with thiocyanogen (or substances yielding thiocyanogen), and the products are boiled with alkali or an alkali sulphide to give sulphide dyes. 1-Hydroxy- α -naphthaphenazine, 1:13-dihydroxy- $\alpha\alpha'$ -dinaphthazine, 4:4':4''-trianilino-triphenylmethane, and tolusafranine give sulphide dyes in this way. The method is applicable to substances which are not thionated satisfactorily by the usual sulphide treatment; aromatic amino- and/or hydroxyl groups must be present.

C. HOLLINS.

Manufacture of vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 289,980, 14.2., 12.9., and 29.9.27).—The nitrobenzanthrone of B.P. 12,518 of 1906 (B., 1906, 844) is reduced to aminobenzanthrone and heated with caustic alkali under mild conditions, *e.g.*, potassium ethoxide at 140–150°, to give a green vat dye. Similar or identical dyes are obtained from certain derivatives of the aminobenzanthrone, namely, the bromo-compound prepared by acetylation, bromination, and hydrolysis, the *p*-tolylthiol derivative obtained from this bromo-compound and thio-*p*-cresol, and the methylthiol derivative resulting from methylation of aminothiobenzanthrone (a by-product of the sodium sulphide reduction). The green dyes may be oxidised, in substance by chromic-acetic acid, or on the fibre with bleaching powder, to give a fast brown vat dye. A similar brown dye is obtained by heating the nitrobenzanthrone with alcoholic alkali at 140°, and may be purified by oxidation with hypochlorite; this brown dye becomes reddish-orange when chlorinated on the fibre. C. HOLLINS.

Vat dye preparation and utilisation. R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 289,103, 13.10.26).—The 3-chloroaminobenzanthrone, m.p. 280–281°, obtained by reducing 3-chloronitrobenzanthrone (B.P. 256,281; B., 1926, 868) is heated with an alkali (alcoholic alkali or an alkali alkoxide), preferably in presence of a thiophenol (*e.g.*, thio-*p*-cresol), to give a vat dye yielding reddish-blue dyeings becoming bright blue when treated with bleaching

powder. The vat dye may be purified by fractional precipitation from sulphuric acid. The process of B.P. 254,742 (B., 1927, 550) is disclaimed.

C. HOLLINS.

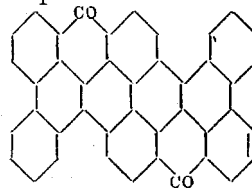
Dyes and dyeing [soluble vat dye derivatives]. B. WYLAM, J. E. G. HARRIS, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 290,690, 17.12.26).—A vat dye (dimethoxydibenzanthrone, indigo, flavanthrone) is treated with a metal (zinc) in boiling pyridine or other tertiary base in presence of an alkyl halide (ethyl bromide), or acid halides (benzoyl chloride), or acid anhydrides (acetic, phthalic). The products are insoluble in water, soluble in pyridine. Some are readily hydrolysed by sulphuric acid, regenerating pure vat dyes. A few may be fixed on the fibre by acid oxidation.

C. HOLLINS.

Manufacture of new vat dyes. I. G. FARBENIND. A.-G. (B.P. 273,247, 7.3.27. Ger., 22.6.26).—The higher alkyl ethers of hydroxylated dibenzanthrones or isodibenzanthrones have poor light-fastness properties. This disadvantage is not shown, however, by the β -alkoxyethyl ethers even when the alkoxy-group contains several carbon atoms. The new derivatives, obtained best by alkylation with the appropriate toluenesulphonic ester, are surprisingly soluble in benzene etc. and are useful both as vat dyes and as pigments for cellulose ester lacquers. The β -methoxyethyl ether of dihydroxybenzanthrone gives fast blue-green dyeings on cotton; the β -*n*-butoxyethyl ether is a green pigment. Glycol ditoluenesulphonate present in β -*n*-butoxyethyl toluenesulphonate gives a blue vat dye (easily separable from the main product), which is also obtainable from dihydroxydibenzanthrone and ethylene dibromide.

C. HOLLINS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G. (B.P. 282,629, 16.9.27. Ger., 23.12.26).—5:13-Diphenylpyranthrone, treated with acid condensing agents (sulphuric acid and manganese dioxide, or aluminium chloride followed by air oxidation), gives a bright orange vat dye of probable constitution



C. HOLLINS.

Manufacture of new [vat and acid] dyes. BRIT. DYE STUFFS CORP., LTD., J. BADDILEY, P. DOOTSON, A. SHEPHERDSON, and S. THORNLEY (B.P. 289,188, 8.2.27).—An aminoflavanthrone is condensed with a polynuclear aromatic halogen compound having a ring-system of at least three fused nuclei, such as halogenated acenaphthaquinones, flavanthrones, pyranthrones, etc., to give vat dyes or, if carboxyl or sulphonic groups are present, acid dyes. *E.g.*, aminoflavanthrone (from flavanthrone and hydroxylamine) is condensed with dibromopyranthrone for a brown vat dye.

C. HOLLINS.

Manufacture of new [vat] dyes. SOC. CHEM. IND. IN BASLE (B.P. 262,774, 7.12.26. Switz., 8.12.25).—Violanthrone, trichlorinated in nitrobenzene at 135–140°,

yields a navy blue vat dye fast to water (cf. B.P. 22,519 of 1905; B., 1906, 634).
C. HOLLINS.

Manufacture of vat dyes. BRIT. DYESTUFFS CORP., LTD., H. M. BUNBURY, H. EVANS, and A. SHEPHERDSON (B.P. 289,191, 9.2.27).—Dyes of increased tinctorial powder are obtained from dibenzoyl-4:4'- and 4:5'-diamino-1:1'-dianthrimides and sulphuric acid by adding an oxidant (perborate, persulphate, or hypochlorite) to the reaction mixture, or treating the reaction product with an oxidant.
C. HOLLINS.

Manufacture of new azo dyes which are insoluble in water. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 290,733, 18.2.27).—A 2:7-diaminocarbazole is tetrazotised and coupled with components other than β -naphthol, either on the fibre or other substrate or in substance. Examples are: 2:7-diaminocarbazole with 5-hydroxy- β -naphthacarbazole (from 2:7-dihydroxynaphthalene; black), or bisacetoacetylolidine (yellowish-brown), or 2:3-hydroxynaphthoic anilide (navy-blue); 2:7-diamino-3:6-dimethylcarbazole with 2:3-hydroxynaphthoic α -naphthylamide (bluish-black); 3:6-dichloro-2:7-diaminocarbazole with 2:3-hydroxynaphthoic anilide.
C. HOLLINS.

Manufacture of new mordant disazo dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 289,135, 18.1.27).—A disazo dye of the type: *p*-phenylenediamine (or homologues or substitution products) = 2 mols. of salicylic acid (or derivatives having 3- and 5-positions unsubstituted), is sulphonated, e.g., with 20% oleum at 130–135°, to give a chrome-printing yellowish-orange dye of especially good fastness to light, soap, and chlorine.
C. HOLLINS.

Manufacture of new [thio]indigoid dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 289,154, 21.1.27).—A halogenated $\beta\beta$ -naphthisatin anil or chloride is condensed with a monoether of 1:4-dihydroxynaphthalene to give green dyes fast to chlorine. Examples are the 2-chlorides of 9-chloro-6- (or 7)-bromo- $\beta\beta$ -naphthisatin, its nitration product, and 9-chloro- $\beta\beta$ -naphthisatin, condensed with 4-methoxy- α -naphthol.
C. HOLLINS.

Production of dyestuff emulsion and of colour lake therefrom. C. E. J. GOEDECKE, Assr. to W. EBERLEIN (U.S.P. 1,672,454, 5.6.28. Appl., 8.5.25. U.K., 16.8.24).—See B.P. 241,332; B., 1925, 933.

Manufacture of [vat] dyes [from polyhalogeno-violanthrones]. SOC. CHEM. IND. IN BASLE (B.P. 263,826, 24.12.26. Switz., 24.12.25).—See U.S.P. 1,659,145; B., 1928, 293.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Production of strong threads from silk coagula. P. P. VON WEIMARN (Kolloid-Z., 1928, 45, 36–39).—Strong fibres of silk are produced when the viscous, highly concentrated solution formed by heating silk in aqueous solutions of very soluble salts (e.g., sodium thiocyanate) is poured into concentrated solutions of coagulating salts such as sodium citrate and potassium sodium tartrate. The threads gradually become brittle on keeping, but a portion can be kept for more than a year

without change. This is discussed in regard to the factors affecting the brittleness of natural silk threads.

E. S. HEDGES.

Properties of silk coagula formed by pouring colloidal silk solutions into concentrated tannin solutions. P. P. VON WEIMARN (Kolloid-Z., 1928, 45, 39–42).—Natural silk readily adsorbs tannin, which can be removed only with difficulty by treatment with water. Coagula containing both fibroin and tannin were made by dispersing silk in concentrated aqueous solutions of very soluble salts (e.g., sodium iodide) and pouring into a 30–40% solution of tannin. The consistency of the coagulum is such that it can be drawn into threads exceeding 1 m. in length, and can be spread into a transparent film covering a large area. On drying the film becomes cloudy, but later is transparent once more and becomes brittle. The coagulum dissolves to a colloid when boiled in concentrated tannin solution, and is regenerated by addition of water. If the disperse system is cooled to the ordinary temperature without addition of water it separates into two layers, and on keeping gives a sticky, viscous mass. The coagulum also disperses in a large volume of boiling soap solution but is not dispersed by weak tannin solutions or by water. In boiling water threads of the coagulum are broken up into small pieces, which in time lose the greater part of their adsorbed tannin. The strength of the threads increases with progressive washing out of the tannin with cold water. Solvents which can remove the tannin from the coagulum without affecting the fibroin aid in the production of strong threads.

E. S. HEDGES.

Detection of damaged wool. W. SIEBER (Textilber., 1928, 9, 326–328).—Mechanical and chemical damage in wool may be detected by immersing the wool in a boiling 1% aqueous solution of benzopurpurin 10B for a few minutes, then washing with boiling water until no further colour is removed from the wool, and examining under a microscope; undamaged raw wool remains colourless, mechanically damaged portions become pink, and wool deteriorated by treatment with an alkali or acid becomes yellowish-pink to red and bluish-pink to red, respectively. The colour changes are more evident in ultra-violet light than in daylight.
A. J. HALL.

[Method for] distinguishing raw and bleached cotton fibres in yarns spun from mixtures of the same. W. SIEBER (Textilber., 1928, 9, 404–406). Bleached cotton is much more easily wetted than raw cotton; it also becomes pink when immersed in a cold solution of benzopurpurin, whereas raw cotton under similar conditions remains colourless. Raw cotton differs from bleached cotton in having a strong affinity for Victoria Blue B; a similar but less marked difference is shown towards Magenta and Malachite Green. The presence of raw cotton in a mixed yarn is detected by immersing the yarn (about 0.1 g.) for $\frac{1}{2}$ –1 min. in 10 c.c. of a boiling solution containing 3% (calc. on wt. of yarn) of Victoria Blue B, then washing with cold water, immersing for $\frac{1}{2}$ –1 min. in boiling water, washing in cold water, and drying; the raw fibres appear uniformly coloured, whereas the bleached fibres are but slightly stained, and the lumen remains uncoloured.

except in isolated places. It is suggested that this test may be helpful in determining the efficiency of a bleaching process and also in investigating the fine structure of plant fibres.

A. J. HALL.

Bemberg [cuprammonium] silk. TEDE (Textilber., 1928, 9, 230—232).—Bemberg silk is manufactured by forcing a cuprammonium solution of cellulose (prepared from cotton linters) through orifices, drawing (with stretching) the resulting filaments through water, treating with sulphuric acid, washing, and winding the silk in hank form; bleaching is not necessary when cotton is used as a raw material. Bemberg silk filaments have a denier not exceeding 1.25 and their cross-sections are circular, but not irregular as with viscose silk; it is softer and has a more silk-like lustre than viscose silk. Bemberg silk becomes deep blue and viscose silk red when dyed for 5 min. in a cold solution consisting of 20 c.c. of a 0.5% solution of eosin, 15 c.c. of pelican ink (No. 4001, G. Wagner), and 65 c.c. of water. Bemberg, viscose, Chardonnnet, and cellulose acetate silk yarns of 100 denier have dry and wet strengths of 200 and 100, 150 and 45—50, 150 and 50—80, and 125 and 75, respectively. Commercial Bemberg silk is usually sized with oil, and this should be removed before dyeing by treatment in a liquor at 60° containing 5 g. of soap, 5 g. of ammonia, and 2% (calc. on wt. of silk) of Nekal AEM per litre. Turkey-red oil must not be used as an assistant in dyeing Bemberg silk since it promotes unevenness ("Finkenbildung"). The strong affinity which Bemberg silk has for basic dyes should be reduced by soaping the silk immediately after mordanting with tannic acid and tartar emetic preparatory to dyeing.

A. J. HALL.

Cellulose acetate and its solutions. I. Composition of cellulose acetate lacquer for aeroplane dope. K. ATSUKI and R. SHINODA. **II. Stability of cellulose acetate.** K. ATSUKI. **III. Stabiliser for cellulose acetate.** Y. TANAKA and K. ATSUKI. **IV. Acetylation of cellulose.** **V. Relation of temperature and time of ripening to viscosity of cellulose acetate.** K. ATSUKI and R. SHINODA (Rept. Aeronautical Res. Inst. Tokyo, 1928, 3, 49—68, 71—88, 91—100, 103—111, 115—123).—I. The examination of various mixed solvents for cellulose acetate by determinations of the solvent power, viscosity of the cellulose acetate solution, and the tensile strength and elongation of the film produced on drying, proves that that solvent which has the highest solvent power yields a solution of minimum viscosity and a film of maximum strength and elongation. Using the most common solvents, the optimum composition is acetone 60%, ethyl alcohol 15—17%, benzene 19—21%, benzyl alcohol or triacetin 1—2%, triphenyl phosphate 1—2%; and the optimum concentration of cellulose acetate is 7—9%. With acetone-ethyl alcohol mixtures minimum viscosity is obtained when the content of alcohol is about 17%; with acetone-benzene mixtures the optimum point is at 20% of benzene; whilst with acetone-alcohol-benzene mixtures it is at 10% of alcohol and 30% of benzene. Addition of triacetin, benzyl alcohol, and triphenyl phosphate tends to increase the viscosity of solvent and solution, and the solvent power. Solutions

containing more than 8% of cellulose acetate show appreciable plasticity.

II. Spontaneous decomposition of cellulose acetate is accelerated by sulphuric acid, which may be present in the incompletely purified product in the free state, as an adsorption compound, or as a cellulose ester, since it is used as a catalyst in the acetylating bath. The amount of sulphuric acid retained decreases as the ripening of the cellulose acetate proceeds, owing to hydrolysis of the sulphuric ester and the more ready diffusion of acid through the material. Details are given for four suggested stability tests for cellulose acetate, viz: 100° heat test, decomposition and carbonisation temperature determinations, and an acetic acid evolution test.

III. Owing to the presence in cellulose acetate of traces of cellulose sulphuric esters, it is desirable to add a stabiliser such as calcium naphthenate. This stabiliser reacts with the sulphuric esters present yielding naphthenic acid, which, like the calcium salt, is a gelatiniser and improves the mechanical properties of the cellulose acetate film. The amount of stabiliser required is usually 1—2%.

IV. On acetylation the cellulose fibre at first swells uniformly, and then locally. The swollen parts gradually disperse into the bath, and the residual short fibres slowly disappear. The triacetate is formed directly without intermediate formation of a mono- or di-acetylated product. If the acetylation is prolonged, the acetic acid content of the ester falls to a minimum and then increases. The most uniform material is obtained by continuing the acetylation until the solubility in acetone is a minimum. The velocity of acetylation is doubled by a rise of 10° between 30° and 50°, but above 50° a rise of over 20° is required to produce the same effect, since decomposition of the cellulose disturbs the relation between velocity increase and temperature.

V. The degree of ripening of cellulose acetate may be followed by determinations of viscosity, which varies with the time and temperature of ripening. The experimental results obtained may be expressed by the equation $T = ae^{kz}$, where T is the temperature, z the time of ripening required to obtain a given viscosity, and a and k are constants; but as the viscosity decreases the temperature range through which this formula holds also decreases.

W. J. POWELL.

Cellulose ester solvents. H. WOLFF [with G. ZEIDLER and W. TOELDTE] (Farben-Ztg., 1928, 33, 2228—2232, 2301—2304).—In dealing with cellulose ester solvents in connexion with lacquer manufacture, considerable misapprehension has been caused by confusing "solvent power" of a solvent with the more important factor of "power to withstand dilution by non-solvents without precipitation of the nitrocellulose." This dilution factor should be correlated with a definite end-concentration of nitrocellulose in the solution, results being obtained indirectly by graphical methods exemplified in the present communication by the behaviour of solvents of the glycol type with respect to various diluents and qualities of nitrocellulose. In this respect as well as in considering viscosity of solution and rate of evaporation of solvent, it is shown

that the influence of non-solvent and of concentration of solution must be taken into account if results of any practical value in lacquer technology are sought. The relative softening effect of solvents on oil undercoats is also discussed. S. S. WOOLF.

Lignin. R. WIGGINTON (Fuel, 1928, 7, 268—272).—An account is given of recent work on the chemistry of lignin; Kürschner's investigations are described in some detail. A. B. MANNING.

Cellulose in peat. HESS and KOMAREVSKY.—See II.

PATENTS.

Carroting or preparation of hair for making it capable of being fulled or felted. E. BÖHM (B.P. 285,028, 21.1.28. Austr., 9.2.27).—Hair is treated in a neutral or acid solution containing hydrogen peroxide and a salt, not of mercury, which is capable of acting as an oxygen conveyor to the hair. Suitable substances are the water-soluble salts of iron, nickel, cobalt, lead, zirconium, bismuth, manganese, cadmium, cerium, copper, chromium, molybdenum, aluminium, and tungsten. *E.g.*, the hair of skins is wetted with a solution containing 6—10% of hydrogen peroxide (or 5—10% of nitric acid and 2—5% of hydrogen peroxide) and 1—2% of an oxygen carrier, then dried at 70—100°, and subjected to the usual after-treatments. A. J. HALL.

Chlorination of hair and wool and of articles made therefrom. E. BÖHM (B.P. 281,646, 24.11.27. Austr., 1.12.26).—Chlorination is effected by nascent chlorine produced in predetermined quantity, if desired by the action of oxidising agents on a solution containing a chloride and an acid. D. J. NORMAN.

Treatment of cotton fabric. L. LILJENFELD, Assee. of H. I. HUEY (B.P. 274,860, 18.7.27. U.S., 20.7.26).—A wool-like character is imparted to cotton fabric by treating the bleached or unbleached material under conditions which allow free shrinkage in both directions first with carbon disulphide (gaseous, liquid, or in solution in, *e.g.*, benzene) and then with caustic alkali solution of not less than 12% strength. Alternatively, an emulsion of 12—20% caustic alkali solution with about 30% of carbon disulphide may be used. After a suitable time, which may vary from a few minutes to several hours, the fabric is washed with water, optionally after treatment with a dilute acid solution, and dried. D. J. NORMAN.

Treatment of yarns and fabrics. OBERRHEINISCHE HANDELSGES. M.B.H. (B.P. 266,367, 17.2.27. Ger., 17.2.26).—Yarns or fabrics of cotton, wool, artificial silk, etc. show increased strength and softness and a reduced gloss and thermal conductivity if they are treated in a wet or dry condition with, *e.g.*, kieselguhr, silica gel, chalk, etc. preferably in the presence of soap solutions, oils, etc. D. J. NORMAN.

Cellulose derivatives for use in the manufacture of artificial textile threads, plastic and like products. SOC. LYONNAISE DE SOIE ARTIFICIELLE, and P. CHEVALET (B.P. 266,300, 10.1.27. Fr., 22.2.26).—Acetoformates of hydrocellulose are prepared by treating cellulose at a low temperature first with formic acid in admixture with less than 50% by wt. of sulphuric

acid (d 1.842), and then with acetic acid mixed with more than twice its weight of sulphuric acid. When the desired degree of esterification has been attained, the product is precipitated in water or alcohol. The resulting ester has all the plastic qualities of cellulose acetates, and is soluble in formic acid up to a dilution of 40%, formaldehyde and its polymerides and derivatives, pyridine, and ureas, cyanates, thiocyanates, calcium chloride, etc. The strength of threads or films made from these esters can be considerably increased by soaking them in a bath of 10% ammonium carbonate solution. D. J. NORMAN.

Manufacture or treatment of cellulose esters. BRIT. CELANESE, LTD. (B.P. 273,743, 1.7.27. U.S., 2.7.26).—The viscosity of cellulose esters can be reduced by treating the ester in suspension or solution with hydrogen peroxide and an iron salt, *e.g.*, ferrous acetate. In the case of cellulose acetate acetylation may be effected in an iron vessel, or iron salts may be added to the acetylating mixture, the hydrogen peroxide being subsequently added when acetylation is complete. D. J. NORMAN.

Manufacture of cellulose esters and conversion products therefrom. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 289,973, 8.2.27).—A halogen derivative of sulphurous acid, *e.g.*, thionyl chloride, and a heavy-metal salt which would of itself act as a catalyst are used conjointly as the catalytic agent in the esterification of cellulose. *E.g.*, 25 pts. of cotton cellulose can be completely acetylated in about 6 hrs. by using a mixture containing, by wt., 75 pts. of acetic anhydride, 100 pts. of glacial acetic acid, 0.25 pt. of zinc chloride or cuprous chloride, and 1.4 pts. of thionyl chloride at 65°. D. J. NORMAN.

Manufacture of artificial [silk] filaments. O. SILBERRAD (B.P. 289,233, 23.3.27).—In the manufacture of artificial silk by the dry-spinning process the difficulties arising from the inrush of air at the point where the filaments leave the spinning chamber may be obviated by the provision of a liquid seal. D. J. NORMAN.

Manufacture of artificial silk. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 289,942, 2.2.27).—Cuprammonium artificial silk, particularly that produced by stretch-spinning, shows increased strength and elasticity if free carbon dioxide or a bicarbonate, *e.g.*, 2 g./litre of sodium bicarbonate, be added to the water into which the filaments are spun. The use of carbon dioxide in the spinning water also minimises the deposition of copper salts on the walls of the spinning funnel. D. J. NORMAN.

Conversion of carbohydrates. R. A. KOCHER (U.S.P. 1,670,727, 22.5.28. Appl., 5.2.20. Renewed 21.3.25).—Cellulosic material is mixed with hydrochloric acid in one container, and this mixture is then transferred to a second container where it is treated with gaseous hydrochloric acid under pressure. The product is transferred to a third container and heated, the hydrochloric acid from this operation being led back for the treatment of a further quantity of raw material. Access of air is avoided throughout. D. J. NORMAN.

Stencil sheets. S. HORII (B.P. 289,511, 24.1.27).—Alcohols derived from oils and waxes of marine animals,

e.g., oleyl and selachyl alcohol (from sperm oil) and cetyl alcohol, are incorporated with esters of polysaccharides in the manufacture of stencil sheets by the process of B.P. 250,798 (B., 1926, 534). D. J. NORMAN.

Recovery of the paste, and in particular wood pulp, from printed paper by the removal of the ink therefrom. L. GRENAUDIER (B.P. 279,516 and Addn. B.P. 282,829, [A] 25.10. and [B] 30.12.27. Fr., [A] 25.10. and [B] 30.12.26).—(A) The paper is shredded and agitated with a boiling 0.1% soap solution until the scum, which must be removed as fast as it is formed, becomes almost white. After rinsing with water, the material is opened up in a beater and the resulting pulp agitated with a boiling alkaline solution, preferably sodium carbonate solution (1:1000). Meanwhile the mixture is stirred, skimmed, and gradually diluted with hot water so that on completion of the process, as indicated by the scum becoming white, the dilution is 1:1500. The pulp is then washed with water and, if necessary, lightly bleached. (B) The pulp is agitated with boiling dilute sodium carbonate (1:800), and suitable reagents, *e.g.*, metal salts or acids, are added to promote scum formation. If desired, the alkaline solution may contain soaps, printing ink solvents, products, such as gelatin, glucose, etc., which favour the formation and separation of scum, loading agents, or bleaching agents.

D. J. NORMAN.

Preparation of modified cellulose for use in the manufacture of cellulose acetate. M. CUSIN, P. A. A. CHEVALET, and SOC. LYONNAISE DE SOIE ARTIFICIELLE (U.S.P. 1,671,513, 29.5.28. Appl.; 7.1.27. Fr., 11.1.26).—See B.P. 264,181; B., 1928, 258.

Manufacture of artificial silk or the like. J. L. RUSHTON (U.S.P. 1,671,785, 29.5.28. Appl., 22.9.26. U.K., 29.10.25).—See B.P. 264,929; B., 1927, 248.

Manufacture of artificial threads etc. C. F. TOPHAM, Assr. to COURTAULDS, LTD. (U.S.P. 1,671,878, 29.5.28. Appl., 30.7.27. U.K., 11.10.26).—See B.P., 278,881; B., 1927, 935.

Production of proofed paper. SCUTAN CO. (B.P. 281,316, 24.11.27. U.S., 26.11.26).—See U.S.P. 1,667,691; B., 1928, 444.

Products from carbohydrates (B.P. 290,377).—See III.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Primary action of chromic acid on wool fibre. M. ILWINSKY and D. KODNER (Z. angew. Chem., 1928, 41, 283—285).—Wool fibre absorbs chromic acid from solution, even in presence of mineral acids, to form a complex in which the wool substance acts as a base, combining with about 10% of chromic acid. This is about the equivalent quantity on the assumption that the molecule contains two basic (NH_2) groups. The complex is stable towards water.

S. I. LEVY.

Indigo losses in the indigo-vat. M. TSCHILIKIN (Textilber., 1928, 9, 318—322).—Indigo losses of 4.38 and 7.17% were observed when air free from carbon dioxide was bubbled through a caustic soda-hyposulphite indigo vat maintained at 45° for $\frac{1}{2}$ and 3 $\frac{1}{2}$ hrs., respectively, these losses differing considerably from

those previously observed. Under similar conditions the loss of thioindigo was 10.97—12.84%. Hydrogen peroxide is formed when air is passed through an indigo vat, and the resulting loss of indigo is due to the action of the hydrogen peroxide on the keto- (not the enolic) form of the indigo-white present, whereby equimolecular quantities of isatin and dioxindole are first formed, the last-named substance being then further oxidised to isatin. The loss of indigo due to the formation of isatin is thus dependent on the relative proportions of the enolic and keto-forms of the indigo-white present. The formation of isatin is not due to the oxidising action of hydrogen peroxide on indigo, or dehydroindigo, or by intermediate formation of indoxyl by excessive reduction as suggested by Crowther (B., 1911, 799).

A. J. HALL.

Raw and bleached cotton fibres. SIEBER. Bem-berg silk. TEDE.—See V.

PATENTS.

Dyeing of pelts, hairs, feathers, etc. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 290,126, 30.11.27).—The pelts, hairs, feathers, etc., with or without mordanting, are treated with a *p*-aminophenyl-naphthylamine salt, which is then oxidised. *p*-Aminophenyl- β -naphthylamine gives a blue-green, or with a copper mordant a black-blue; the α -isomeride produces red-violet to corinth shades. Other examples are 2-*p*-aminoanilino-6-naphthol (grey), 2:6-bis-*p*-aminoanilinonaphthalene (greenish-grey), 1:5- or 2:7-isomeride (violet-grey), 1-*p*-aminoanilino-5-naphthol (blue-violet), 2:7-isomeride (blue-violet). The phenyl group may carry an alkyl or halogen substituent.

C. HOLLINS.

Dyeing of fabrics containing previously immunised cotton fibres mixed with other fibres. SOC. ANON. DES ÉTABL. PETITDIDIER [ANC. MAISON JOLLY-BELIN] (B.P. 268,327, 15.3.27. Fr., 29.3.26).—The process of B.P. 261,399 (B., 1928, 365) is applied to the dyeing of fabrics composed substantially of immunised cotton.

C. HOLLINS.

Dyeing of esters and ethers of cellulose and its conversion products. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 290,720, 16.2.27).—Acetate silk (etc.) is dyed from a neutral or weakly acid bath with azo dyes of the type: dinitroaminophenol \rightarrow aminonaphthol. Examples are: 4:6-dinitro-*o*-aminophenol with 1:8-aminonaphthol (a water-soluble black dye), or with 1:6-, 2:8-, and 1:5-aminonaphthols for brown shades, intensified by diazotisation on the fibre and development; 2:4-dinitro-6-amino- and 1:7-aminonaphthol for a violet-brown dyeing which may be diazotised and developed with β -naphthol (brown-black), ethyl- β -naphthylamine (green), or 2:4-tolylene-diamine (brown).

C. HOLLINS.

Production of fast tints on cellulose esters or ethers. SOC. CHEM. IND. IN BASLE (B.P. 281,704, 2.12.27. Switz., 4.12.26).—Acetate silk is dyed with a diaminoazo dye (*e.g.*, hydrolysed *p*-aminoacetanilide \rightarrow cresidine) in presence of a dispersing agent, and then diazotised and developed in weak acid medium with 2:3-hydroxynaphthoic acid (deep black) or β -naphthol

(deep black). Navy-blue shades are obtained by using less diaminoazo compound. C. HOLLINS.

Wetting and degreasing agents [for textiles]. G. ZIMMERLI (U.S.P. 1,672,292, 5.6.28. Appl., 15.6.25. Switz., 27.6.24).—See B.P. 236,209; B., 1926, 404.

Apparatus for printing fabrics in several colours. M. RATIGNIER (B.P. 277,320, 30.8.27. Fr., 11.9.26).

Vat dye (B.P. 289,103). Soluble vat dye (B.P. 290,690).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Ammonia synthesis with catalysts obtained from complex cyanides of iron. A. MITTASCH and E. KUSS (Z. Elektrochem., 1928, 34, 159—170).—In a catalyst prepared from potassium aluminium ferrocyanide the presence of free iron, iron carbide and nitride, free carbon, and aluminium carbide, nitride, and oxide was established. By a characterisation of the ferromagnetic portion of the catalyst it is shown that the free iron is present as α -iron. The active catalyst is free iron and not iron carbide; the presence of the latter even after prolonged use may be due to the attack of the surface of carbide particles by hydrogen, yielding a protective coating of iron. The complex cyanides in producing the catalyst may either be reduced or pass through a series of decompositions giving: $\text{Fe}(\text{CN})_2 \rightarrow \text{"carbide X"} + \text{N}_2 + \text{iron nitride}$; $\text{"carbide X"} \rightarrow \text{Fe} + \text{C}$ and $\text{Fe}_3\text{C} + \text{C}$. Thus the formation of Fe_3C without simultaneous production of metallic iron cannot take place. H. INGLESON.

Determination of ammonia. K. TÄUFEL and C. WAGNER (Z. angew. Chem., 1928, 41, 285—287).—The details of manipulation and of the precautions to be taken in the determination of small quantities of ammonia, in presence of nitrogenous materials readily decomposed with formation of ammonia, are discussed for the methods of blowing through large quantities of air and of distilling with excess of magnesium oxide. In the latter method low results are obtained if the solution to be distilled contains a high proportion of magnesium salts. S. I. LEVY.

Plasticity measurements on milk of magnesia. S. ARZOOMANIAN (J. Amer. Pharm. Assoc., 1928, 17, 443—449).—Data and results from plasticity measurements on milk of magnesia are submitted as a practical application of the burette consistometer (cf. Herschel and Bulkley, B., 1927, 127). No definite empirical relationships between yield value or mobility or the settling or assay of a milk were found. The penetrativity method indicated that milk of magnesia is a plastic with a low yield value and high mobility. Flow-pressure curves are given and the average pressure, surface tension, and kinetic energy corrections are discussed. E. H. SHARPLES.

Comparison of the methods of determining potassium ferrocyanide. P. P. BUDNIKOV (Z. anal. Chem., 1928, 73, 433—438).—Volumetric determination of ferrocyanide by precipitation with solutions of zinc or silver salts gives erratic results due to the difficulty of ascertaining the end-point, the iodometric method

gives high results and is influenced by a time factor, and the gravimetric methods are too tedious for technical purposes. Accurate results are obtained by titration with permanganate provided that the ferrocyanide solution is more dilute than 0.05N. A. R. POWELL.

Evolution of acetylene from calcium carbide by the action of water, hydrogen sulphide, and hydrogen chloride, in the liquid and gaseous conditions. E. BIESALSKI and H. VAN ECK (Z. angew. Chem., 1928, 41, 278—282).—The extent of the gas evolution depends on the extent to which the reacting vapour is absorbed by the carbide, and on the degree to which the solid products formed protect the remaining carbide from further attack. Dry steam decomposes 20% of the carbide present in 2 hrs. at 130°; at 450° there is no attack. This is due to the formation of carbon by dissociation of acetylene, the carbon protecting the carbide from further attack. Hydrogen sulphide and chloride both react exceedingly slowly in the gaseous state; even the liquefied gases react very slowly, since in both cases the solid products protect the carbide from further attack. S. I. LEVY.

Acetic acid and its derivatives. TERLINCK.—See III. **Analysis of phosphate rock.** REYNOLDS and others, also JACOB and REYNOLDS.—See XVI.

PATENTS.

Manufacture of chromic acid in a solid form soluble in water. R. SCHUSTER (B.P. 291,249, 23.6.27).—A mixture of chromium trioxide or sodium or potassium dichromate, sulphuric acid, sodium and/or potassium silicate, and water is concentrated until a test portion sets to a crystalline mass on cooling. L. A. COLES.

Production of compounds of nitrogen and oxygen from ammonia. N. CARO and A. R. FRANK (B.P. 274,099, 7.7.27. Ger., 8.7.26).—Gaseous mixtures of ammonia and air or oxygen, together with hydrogen, are subjected to catalytic combustion under pressure according to the method described in B.P. 273,718 (B., 1923, 402). W. G. CAREY.

Manufacture of ammonia. SOC. D'ÉTUDES MINIÈRES & INDUSTRIELLES (B.P. 274,023, 14.1.27. Fr., 6.7.26. Addn. to B.P. 253,540; B., 1927, 218).—Alkali or alkaline-earth halides, oxyhalides, oxides, ferrocyanides, or ferricyanides, separately or in mixture, are used instead of alumina, magnesia, or lime, thus lowering the reaction temperature to 380—450°. The lithium amide employed has the formula Li_3NH_2 . W. G. CAREY.

Apparatus for use in the manufacture of alkali and alkaline-earth hydroxides. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 290,719, 16.2.27).—Amalgams of alkalis or alkaline earths are stirred or propelled in water by means of charcoal, coke, or graphite stirrers or scoop wheels, or such carbonaceous material mixed with an alloy chiefly containing iron, chromium, or nickel. W. G. CAREY.

Recovery of sodium nitrate. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 290,860, 21.7.27).—Sodium nitrate is recovered from solution, e.g., that obtained in the treatment of caliche, by adding sodium

sulphate to form with the nitrate a double salt sparingly soluble at ordinary temperature, separating the double salt, and heating it at above 70° with just sufficient water or dilute nitrate solution to dissolve the sodium nitrate and leave the sodium sulphate. W. G. CAREY.

Manufacture of alkali iodates. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 290,749, 22.2.27).—Oxygen is caused to react on (a) a melt of 3 pts. of caustic alkali and 1 pt. of alkali iodide at 400° and 5–15 atm., or (b) well-stirred alkali iodide at above 400° and 100 atm., or (c) a solution of alkali iodide above 100° and 1 atm. in presence of a catalyst providing hydroxyl ions, *e.g.*, caustic soda or caustic potash lye.

W. G. CAREY.

Preparation of ammonium sulphate. S. ROBSON (B.P. 289,950, 3.2.27).—Ammonia, sulphur trioxide direct from the contact plant, and water are allowed to react together in the form of vapour or mist, and the resulting ammonium sulphate is collected in settling chambers and in a bag-filtration plant. The ammonia and sulphur trioxide may be mixed together in a reaction chamber and combination brought about by the introduction of steam or a fine spray of water, or either of the gases may be introduced together with moisture into an atmosphere of the other.

A. R. POWELL.

Electrolytic manufacture of compounds containing active oxygen. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 290,750, 22.2.27).—Per-compounds are produced simultaneously at the anode and cathode of an electrolytic cell. For example, ammonium persulphate is produced in the anode chamber and hydrogen peroxide in the cathode chamber by using saturated ammonium sulphate in the anode chamber separated by a porous diaphragm from 0.5% sulphuric acid through which a vigorous stream of oxygen is passed in the cathode chamber. Platinum anodes and amalgamated gold cathodes are used. Percarbonate and perborate can also be produced simultaneously.

W. G. CAREY.

Production of manganese peroxide and other manganese oxides from manganese-containing ores and substances. K. E. DITTMANN, K. FAERBER, and GELSENKIRCHENER BERGWERKE A.-G. (B.P. 290,491, 10.9.27).—The material containing manganese is decomposed in a fine dispersive form with dilute sulphurous acid, the resulting manganese sulphate lye is separated, evaporated, and the sulphate roasted with coke and sintered with the admission of air. The processes are conducted in closed vessels to recover the sulphurous acid for use again in the process.

W. G. CAREY.

Catalysts for the catalytic production of methyl alcohol and other oxygenated organic compounds from oxides of carbon and hydrogen. SYNTHETIC AMMONIA & NITRATES, LTD., and R. G. FRANKLIN (B.P. 290,399, 15.11.26).—A mixed zinc-chromium catalyst is prepared by boiling zinc carbonate (precipitated alone or on a carrier) with a solution of chromic acid or its salts. The ratio Zn:Cr in the product is about 9:1 and may be adjusted by washing. If zinc chromate is the chromate used the product is dried in hydrogen or reducing gases.

C. HOLLINS.

Separation of tin and arsenic compounds. W. T. LITTLE (U.S.P. 1,670,307, 22.5.28. Appl., 27.11.25).—A mixture of sodium stannate and arsenate is leached with a liquid sufficiently hot to melt the sodium arsenate.

II. ROYAL-DAWSON.

Manufacture of organometallic compounds [lead and tin tetra-alkyls]. C. ARNOLD. From STANDARD DEVELOPMENT Co. (B.P. 290,444, 21.5.27).—Sodium-lead is treated with ethyl chloride or bromide in a hydrocarbon solvent (naphtha) in a rotating autoclave at 40–70° and finally at 100°; lead tetraethyl is isolated in the usual manner. The process is applicable to the preparation of other lead alkyls and tin alkyls.

C. HOLLINS.

Production of hydrogen from methane hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 291,244, 18.6.27).—Mixtures of methane etc. with steam are passed successively over a catalyst and material capable of absorbing the carbon dioxide formed, *e.g.*, calcium or cerous oxide, at a temperature sufficiently high to prevent condensation of water, the process being repeated if necessary. The oxide is subsequently regenerated by heating the carbonate.

L. A. COLES.

Manufacture of carbon dioxide. P. E. HAYNES (B.P. 277,938, 15.7.27. U.S., 27.9.26).—Limestone is calcined and the carbon dioxide is drawn over limestone subsequently to be calcined to preheat it. Steam which has already operated a compressor for condensing and solidifying the carbon dioxide passes in counter-current to the quicklime formed, hydrating and cooling it, while the excess steam reduces the dissociation temperature of the limestone and takes up the sensible heat and the heat of hydration of the lime to preheat further limestone.

W. G. CAREY.

Catalytic oxidation of sulphur dioxide. H. G. C. FAIRWEATHER. From SELDEN Co. (B.P. 290,316, 4.1.27).—The catalyst comprises an artificial zeolite containing the pentoxides of phosphorus, arsenic, or vanadium, the trioxides of chromium, molybdenum, or tungsten, or manganese heptoxide or dioxide in a non-exchangeable form, together with a catalytically active base, other than vanadium, in an exchangeable form. The catalyst may be diluted with a porous siliceous material, *e.g.*, kieselguhr, or may be coated on to massive granular fragments of ferro-alloys, quartz, ilmenite, or metal oxides. These catalysts are not affected by substances which poison platinum.

A. R. POWELL.

Production of sulphur dioxide-containing gas currents for the manufacture of sulphuric acid. R. VON ZELEWSKI (B.P. 277,382, 13.9.27. Ger., 13.9.26).—The gas mixtures are produced by passing air under pressure downwards through the grate of Dwight-Lloyd or similar apparatus for roasting sulphide ores etc., and are passed directly into the Glover tower without cooling.

L. A. COLES.

Manufacture of phosphorus, phosphoric acid, and hydrogen. COMP. NAT. MAT. COL. & MANUF. PROD. CHIM. DU NORD RÉUNIES, ÉTABL. KUHLMANN (B.P. 286,290, 20.1.28. Fr., 3.3.27).—The condensate from a phosphate reducing furnace is treated with superheated steam at ordinary or reduced pressure

and below the b.p. of phosphorus in a container suitably heated. The latter may be used for cooling and condensing the vapours passing from the furnace to the condenser, the phosphorus being condensed or the vapour used direct to prepare phosphoric acid and hydrogen. W. G. CAREY.

Preparation of catalytic materials [for synthesis of ammonia]. G. CLAUDE, Assr. to LAZOTE, INC. (U.S.P. 1,671,345, 29.5.28. Appl., 21.10.20. Fr., 5.6.28. Appl., 17.9.25. Ger., 13.10.24).—See B.P. 269,344; B., 1927, 524.

Production of iron-free alumina. H. SPECKETER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,672,788, 5.6.28. Appl., 17.9.25. Ger., 13.10.24).—See B.P. 241,184; B., 1926, 51.

Catalytic oxidation of sulphur dioxide. H. G. C. FAIRWEATHER. From SELDEN Co. (B.P. 290,089, 14.9.27).—See U.S.P. 1,660,511; B., 1928, 320.

Evaporation of liquids (B.P. 289,116).—See I. **Ammonium phosphate** (B.P. 290,518). **Fertilisers** (B.P. 282,330, 283,908, and 280,226).—See XVI.

VIII.—GLASS; CERAMICS.

Danger to health of antimonial enamels. B. REWALD (Z. angew. Chem., 1928, 41, 287—288).—A criticism of the conclusions of Flury (B., 1927, 877). Stress is laid on the innocuous character of sodium metantimonate and compounds of quinquivalent antimony generally. In a rejoinder FLURY points out that compounds of quinquivalent antimony may be reduced in the actual process of manufacture of the enamel, and that enamels made from such compounds have in fact been shown to yield salts of tervalent antimony to acid solutions. S. I. LEVY.

Standards Report of the American Ceramic Society (J. Amer. Ceram. Soc., 1928, 11, 334—534).—Definitions, methods of analysis, testing apparatus, standard specifications, etc. are discussed.

PATENTS.

Manufacture of fused silica. QUARTZ & SILICE (B.P. 283,148, 23.12.27. Fr., 5.1.27).—Silica containing no carbon or free silicon is obtained by moistening sand etc. with about 2% of water before it is charged into the melting furnace. L. A. COLES.

Melting of enamels, frits, and glazes. T. J. VOLKKOMMER (B.P. 280,895, 2.11.27).—A rotary furnace for melting enamels etc. in which the geometric axis is inclined to the axis of rotation is provided with a tapping hole at one of the lowest points of the drum which can be reached during the rotation, and with a charging opening arranged axially with respect to the rotation, which also serves as an outlet for the furnace gases. The furnace is mounted on a travelling frame on which it can be transported from the furnace to the charging apparatus. L. A. COLES.

Manufacture of refractory articles of clay. J. HOCHHUT (B.P. 291,305, 12.12.27).—A mixture of clay and "shortening" material moistened with sufficient water to produce a loose coherent mass, with or without the addition of a little oil, preferably formed by mixing the whole of the water with the shortening material

and subsequently incorporating the clay, is introduced in small portions into an open mould, each portion being stamped down before adding the next. The moulded article, containing about 8.8% of water, is dried and fired in the usual way. L. A. COLES.

Kilns for burning clay products, glazed ware, and pottery. H. WEBSTER (U.S.P. 1,671,559, 29.5.28. Appl., 15.10.26. U.K., 17.10.25).—See B.P. 269,344; B., 1927, 524.

IX.—BUILDING MATERIALS.

PATENTS.

Employment of cement and the like. G. N. WHITE, E. G. BECKETT, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 290,309, 10.11.26 and 12.9.27).—The formation of incrustations on the surface of cement and concrete is prevented by the addition to the cement of commercial ammonium carbonate in such quantity that at least 5%, calculated on the weight of the dry cement, remains unattacked by the constituents of the cement. The ammonium carbonate is preferably incorporated as a saturated solution after the cement has been gauged with water. Mineral pigments or vat dyes, e.g., Caledon brilliant purple RK, Caledon red BN, or Caledon blue RC, may also be incorporated with the mixture. Cement washes may contain 25—50% of ammonium carbonate or the cement may be brushed with saturated ammonium carbonate solution immediately after application. L. A. COLES.

Manufacture of cement. E. C. R. MARKS. From INTERNAT. CEMENT CORP. (B.P. 290,890, 26.9.27).—Volume-constant Portland cement of improved tensile strength is obtained by subjecting normal cement clinker to one or more burning operations after it has been ground and mixed with lime, calcium carbonate, etc., in quantity not exceeding that required for the formation of tricalcium silicate, and, if desired, gypsum. L. A. COLES.

Manufacture of finishing lime. E. O. SCHNELL (U.S.P. 1,670,425, 22.5.28. Appl., 1.2.26).—In making a highly plastic finishing lime, burned limestone is pulverised, hydrated with an emulsion of colloidal clay and earth in water, and to the cooled product is added a further amount of the colloidal mixture in a dry state. H. ROYAL-DAWSON.

Paving materials. F. RASCHIG (Dr. F. RASCHIG) (B.P. 291,276, 20.10.27).—A hard, lumpy material which emulsifies readily with water is prepared by adding molten pitch etc. to a mixture of clay with sufficient water to form an emulsion, e.g., about equal pts. by wt., at 40—50°, and allowing the product to cool. Fillers may also be added. L. A. COLES.

Manufacture of asphaltic material [for roads]. J. D. FORRESTER, Assr. to P. J. MCINERNEY (U.S.P. 1,671,070, 22.5.28. Appl., 26.6.25).—To crushed stone aggregate, previously dried, is added, while still hot, a light oil, the flash point of which is above the drying temperature; the mixture is finally incorporated with an asphaltic binder. H. ROYAL-DAWSON.

Production of road-surfacing material. F. W. VALLÉ-JONES (B.P. 290,323, 10.1.27).

Manufacture of material for road surfaces, floors, etc. F. CARL and C. RIEDEL (B.P. 279,093, 13.10.27. Ger., 15.10.26).

Mixture for use in the construction of cold asphalt road pavements. A. C. BROWN (B.P. 290,725, 17.2.27).

Manufacture of building slabs or panels. S. TCHAYEFF (B.P. 290,827, 17.5.27).

Feeding of muddy material into rotary [cement] kilns and the like. J. S. FASTING (B.P. 284,276, 7.11.27. Denm., 27.1.27).

Preservation of wood. B. R. V. MALENKOVIC (B.P. 263,173, 16.12.26. U.S., 16.12.25).—See U.S.P. 1,656,863; B., 1928, 266.

Preservation of stone, wood, etc. (B.P. 290,331).—See XIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Effect of phosphorus and sulphur in steel.
Effect of sulphur on plate material. G. K. BURGESS and others (Proc. Amer. Soc. Testing Materials, 1927, 27, i, 135—138).—Results of tensile, shear, and bend tests are not systematically related to the sulphur content (up to 0.077%). CHEMICAL ABSTRACTS.

Grain growth in low-carbon steel. H. L. PUBLOW and L. J. WALDRON (Mich. Eng. Exp. Sta. Bull., 1927, No. 9, 30 pp.).—The crystallisation of steel is described by reference to the equilibrium diagram. Samples of steel (0.2% C) of various grain sizes, when heated at 1010° for 1—4 hrs. and slowly cooled, exhibited the same grain size. CHEMICAL ABSTRACTS.

Grain formation in low-carbon steel within the critical range. H. L. PUBLOW and L. J. WALDRON (Mich. Eng. Exp. Sta. Bull., 1927, No. 14, 27 pp.).—Low-carbon steels were normalised at 1010°, then reheated to various temperatures, and slowly cooled. A decrease in grain size was observed with those heated at 832—927°. The changes are discussed in detail.

CHEMICAL ABSTRACTS.

Potentiometric analysis of the hardening elements in special steels. II. **Determination of chromium and vanadium in iron alloys and superior steels.** E. ZINTL and P. ZAIMIS (Z. angew. Chem., 1928, 41, 543—546; cf. B., 1927, 939).—In the determination of chromium and vanadium by the authors' potentiometric method, the presence of iron is not essential. Small quantities of manganese must, however, be present. Tungsten interferes with the determination of vanadium, but not in the presence of phosphoric acid. The sample taken for analysis (0.5—5 g., depending on the chromium and vanadium content) is fused with 6—10 times its own weight of sodium peroxide and, after cooling, extracted with 2*N*-sodium hydroxide. A few c.c. of hydrogen peroxide are added to decompose manganate and ferrate, and the whole is boiled. After filtration, the filtrate, which contains the whole of the chromium, vanadium, molybdenum, and tungsten as chromate, vanadate, molybdate, and tungstate, respectively, is diluted to an appropriate volume. A suitable aliquot is acidified with 100 c.c. of

1 : 1 (vol.) sulphuric acid, 2—3 c.c. of a dilute manganese sulphate solution (120 mg. of crystals/litre) are added, and after dilution to 200 c.c. the titration is effected as described. If tungsten is present, the aliquot is treated with 30 c.c. of phosphoric acid (*d* 1.74) before the addition of the sulphuric acid. The catalytic effect of the small amount of manganese is discussed.

J. S. CARTER.

Development of some fundamentals in the ferric sulphate-sulphuric acid process [for copper ores]. F. S. WARTMAN and H. E. KEYES (U.S. Bur. Mines Rep. Invest., 1927, [2839], 11 pp.).—A study of the oxidation stage of the process of making mixed solutions of ferric sulphate and sulphuric acid suitable for leaching certain low-grade copper ores. CHEMICAL ABSTRACTS.

Dependence of the elastic coefficient of extension (α) of copper on the previous [heat and mechanical] treatment. W. KUNTZE (Z. Metallk., 1928, 20, 145—150).—The coefficient of extension, α , is the reciprocal of the modulus of elasticity, i.e., the increase in unit length caused by unit load. In copper test-pieces that have just previously been subjected to plastic deformation, the value of α is subject to hysteresis with loads up to $\frac{1}{4}$ — $\frac{1}{2}$ of the limit of proportionality in the ordinary tensile test, but increases linearly with the load. With greater loads the value of α increases at a greater rate. The effect of drawing on this value is to cause a rapid increase up to a 3% reduction in area, and then a steady decrease. Tempering at low temperatures to remove internal stress decreases α , and severely drawn and tempered wires give a lower value for α than annealed wires. Storing at the ordinary temperature for long periods has the same effect on α as a low-temperature tempering. A. R. POWELL.

Season-cracking of small-arms cartridge cases [in India] during manufacture. F. S. GRIMSTON (Inst. Metals, March, 1928. Advance copy, 24 pp.).—Bursting of cartridge cases when fired is due to the season-cracking of the brass case during the drawing or after-processes of manufacture. Variation in temperatures of annealing, strength of lubricant on the drawing machines, or contact with soapsuds during storage did not cause cracking, which made its appearance, however, if the cases were stored in contact with even extremely dilute sulphuric acid. More severe internal stresses are caused by a die of conical form than by a bell-shaped die, and cartridge cases formed in a conical die were more prone to season-cracking, a defect which is accelerated also by hot moist climates such as prevail in India during the monsoon.

C. A. KING.

Concentration of Britannia ores. H. A. PEARSE (Trans. Canad. Inst. Min. and Met., 1927, 30, 915—928).—The ore consists of a mixture of chalcopyrite and iron pyrites in a hard quartz-porphry gangue. The constituents being relatively coarse are readily freed from one another by grinding to 60-mesh. During the coarse-grinding stages in gyratory crushers and rolls the slime and fine ore are periodically washed out through screens in order to remove soluble salts which interfere with the subsequent flotation. The final grinding is effected in wet ball-mills using a pulp containing 6.5% of solids, and the discharge after addition of a small

quantity of lime passes directly to the rougher cells in which the sulphide minerals and the gold and silver are separated from the gangue. The flotation agents used are 0.05 lb./ton of potassium xanthate, added partly at the head and partly in the middle of every rougher, and 0.15 lb./ton of a mixture of 80% of coal tar and 20% of Canadian hardwood creosote added at the grinding mills. To produce a stable froth 0.25 lb./ton of steam-distilled pine oil is added with the xanthate, and sometimes small quantities of sodium resinate are added at various points along the roughers. The primary slimes obtained in the early stages of crushing are washed, treated with 8 lb./ton of sodium carbonate, and floated with 0.3 lb./ton of xanthate, 0.15 lb./ton of pine oil, and 0.05 lb./ton of fumol (a condensation product of acetaldehyde). The average concentrate from the rougher cells contains 9–11% Cu; it is classified and the underflow is reground with 1.5–2 lb./ton of slaked lime. The overflow from the classifiers and the discharge from the regrinding mill are refloated with the addition of a little pine oil; the concentrates from this operation average 20% Cu, the pyrite having been deadened by the addition of lime, and the tailings 3% Cu. The latter are retreated in a Forrester pneumatic machine to obtain a middling product with 12% Cu which is re-treated with further quantities of rougher concentrates. The tailings from the Forrester machine are passed over blanket tables to recover free gold, then passed to other Forrester machines in which the pyrite is floated with 0.3 lb./ton of xanthate. The recovery of pyrite is 60% of that in the mill feed, and the product assays 49.8% S, 44% Fe, 0.35% Cu, and 4.5% of insoluble material; the recovery of copper in the first concentrates is 91.3% and the recovery of gold on the blankets about 62%. The cost of treatment of the ore is 42.6 cents/ton, of which 4.5 cents is the cost of flotation reagents and 16.1 cents the labour cost.

A. R. POWELL.

Leaching raw and roasted tin concentrates and tin oxides. C. G. FINK and C. L. MANTELL (Eng. Min. J., 1928, 125, 201–206).—Attempts to extract stannic oxide from ores are described. CHEMICAL ABSTRACTS.

Thermic reduction with metals of Bolivian tin concentrates. C. G. FINK and C. L. MANTELL (Eng. Min. J., 1928, 125, 325–328).—Powdered aluminium, but not iron, lead, bismuth, or copper (present in the concentrates), reduces stannic oxide to tin. The presence of ferric or bismuth oxide markedly reduces the m.p. of stannic oxide. CHEMICAL ABSTRACTS.

Certain alloys of the lead-tin-cadmium system and their use as solders. C. E. SWARTZ (Amer. Inst. Min. Met. Eng. Tech. Pub., 1928, No. 85, 11 pp.).—In general, solders having compositions in the neighbourhood of the ternary lead-tin-cadmium eutectic are best; the addition of 1.5–2.0% of zinc is favourable. CHEMICAL ABSTRACTS.

Thermodynamic study of fundamental corrosion reactions. W. J. SWEENEY (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 11 pp.).—From a critical survey of data available in the literature the following values for the decrease of free energy ($-\Delta F$) of reactions have been calculated: (1) $\text{Fe} + 2\text{H} = \text{Fe}^{++} + \text{H}_2$

(1 atm.); $-\Delta F = 20,350 + 1365 \log (a_{\text{H}^+}/a_{\text{Fe}^{++}})$ g.-cal., where a_{H^+} and $a_{\text{Fe}^{++}}$ are the activities of the corresponding ions; (2) $\text{Fe} + \text{O}_2 + \text{H}_2 = \text{Fe}(\text{OH})_2$; $-\Delta F = 120,900$ g.-cal. at 25°; (3) $\text{Fe} + 2\text{H}_2\text{O} = \text{Fe}(\text{OH})_2 + \text{H}_2$ (1 atm.); $-\Delta F = 7780$ g.-cal. Hence even in the absence of dissolved oxygen there is a small tendency for iron to form ferrous hydroxide. In a solution of given hydrogen-ion concentration the concentration of ferrous ions corresponding to equilibrium of reaction (1) is much greater than can exist if the solution is saturated with ferrous hydroxide. Hence the reactions (1) and (3) cannot be at equilibrium at the same time.

H. J. T. ELLINGHAM.

Metal-spraying process and the prevention of corrosion. R. HOPFER (Chem. Fabr., 1928, 316–318).—The process consists of the melting of a wire in an oxy-hydrogen flame and the atomisation of the molten drops by a current of air. Plated surfaces not more than 0.02 mm. thick and of reasonable strength can be made in this way. It is essential that the droplets should be hot when they strike the surface to be plated, and that the latter should be roughened. All metals can be sprayed which have m.p. below 1600° and are available as wire. The surfaces produced are, however, porous, and the process is only useful when they can be improved by chemical or mechanical means. In the case of iron covered with zinc, the porosity is no disadvantage, as the zinc acts as an electrical protector. Aluminium coatings have found an extended use. The plated material is heated in absence of air, when alloying at the surface of contact takes place and the coating becomes non-porous. A partly alloyed aluminium coating, especially with an additional zinc coating, is a good protection against sulphur dioxide. A more completely alloyed aluminium coat is protective against oxidising gases at temperatures up to 1000°.

C. IRWIN.

Electrolytic production of heavy metals from fused electrolytes. II. G. NEUENDORFF and F. SAUERWALD (Z. Elektrochem., 1926, 34, 199–204; cf. B., 1926, 131).—The preparation of iron, chromium, and manganese from melts of oxides and silicates has been carried out to ascertain how the current yield depends on the temperature and duration of electrolysis. Various difficulties met with in obtaining measurements of the current yield are mentioned, and the advantages of the use of high temperatures are pointed out. The current yields (calculated as Fe^{++}) depend on the duration of electrolysis and on the corresponding changes in concentration in a manner which is not entirely clear. Practically pure carbon-free iron, steel, and pig iron can be obtained under suitable conditions. The iron silicates used suffer loss of oxygen by thermal decomposition. By electrolysis chrome iron ore, iron-chromium-carbon alloys are obtained in high current yields. A mixture of manganese and iron ores gives an alloy very poor in manganese. H. INGLESON.

X-Ray studies of high-speed steel. A. ST. JOHN (Proc. Amer. Soc. Testing Materials, 1927, 27, i, 247–251).

PATENTS.

Roasting furnace. D. BAIRD, Assr. to NICHOLS COPPER CO. (U.S.P. 1,669,925, 15.5.28. Appl., 8.10.26).

—A mechanically rabbled, multiple-hearth roasting furnace contains a hollow revolving shaft carrying on every hearth hollow toothed rabblers supported on annular hollow protuberances on the shaft. Means are provided for circulating cooling water through the shaft and rabblers. A. R. POWELL.

[Dry-ore] concentrator. W. YOUNG, Assr. to M. E. YOUNG (U.S.P. 1,659,935, 21.2.28. Appl., 17.4.26).—The frame of the concentrator is actuated from a rotary shaft mounted in the base. One end of the frame is movably connected to the base, a forked portion at the other end terminates in brackets having a third bracket between them, and an eccentric connexion is provided between each bracket and the shaft. H. HOLMES.

Ore-roasting apparatus. D. W. BOWERS (U.S.P. 1,662,811, 20.3.28. Appl., 6.8.27).—Into the upper part of a vertical, cylindrical retort a mixture of finely-divided ore and high-pressure air is blown at such an angle that the mixture rotates round the walls of the furnace where it meets a stream of fuel directed at right angles to its path. The retort is surrounded with a washing condenser through which the gases pass from the lower end of the retort. A. R. POWELL.

Annealing and heat-treating furnace. A. T. KATHNER (U.S.P. 1,669,902, 15.5.28. Appl., 26.5.26).—The furnace comprises a heating chamber and a cooling chamber in alinement with each other and separated by a refractory wall provided with a communication passage in its lower portion and means for deflecting the flue gases away from the passage. The chambers are provided with a continuous conveyor device, the heating chamber with a damper-controlled stack at its forward end, and the cooling chamber with removable partitions in the roof to facilitate cooling. A. R. POWELL.

Furnace [for heat-treating galvanised sheets]. H. W. THEISS, Assr. to SUPERIOR SHEET STEEL CO. (U.S.P. 1,670,754, 22.5.28. Appl., 23.5.27).—The furnace comprises a heat-treating chamber open at both ends, below which is the combustion chamber provided with flues opening into the upper chamber at the charging end and with an inclined body portion to overlie the galvanising bath. An endless carrier circulates on guide-ways through the furnace and below the combustion chamber. A. R. POWELL.

Casting of ingots. E. C. WASHBURN (U.S.P. 1,670,329, 22.5.28. Appl., 26.3.26).—The bottom and sides of the ingot are cooled by streams of air, but the cooling of the top portion is retarded by applying the heat of the ingot itself. F. G. CLARKE.

Manufacture of steel. J. W. HORNSEY (B.P. 290,317—8, 5.1.27).—In the manufacture of steel in a basic open-hearth furnace by melting sponge iron reduced from iron ore, (A) phosphorus is prevented from entering the steel by leaving a proportion of unreduced iron oxide in the sponge or by adding a proportion of unreduced ore to the sponge, previous to melting, and (B) sulphur is prevented from entering the steel by adding about 5% of limestone to the raw ore before it enters the pre-heater previous to reduction. A. R. POWELL.

Direct production of steel or steel alloys from titaniferous ores and iron sands. F. A. DE SILVA

and C. G. CARLISLE (B.P. 290,442, 18.5.27).—The ore or sand is treated on a magnetic separator and the magnetic product melted at 1600° with 20—33% of anthracite, 7.5% of lime, and 1.5—3.0% of fluorspar in an arc furnace of the type provided with overhead electrodes. The products are pig iron and a fluid slag containing all the titania. The iron is decarburised, refined, and converted into steel or a steel alloy in the same or in an auxiliary electric furnace. During the reducing and refining processes the furnace is rotated or the bath may be rotated relatively to the body of the furnace. A. R. POWELL.

Corrosion-resistant ferrous alloy. J. T. HAY (B.P. 290,487, 5.9.27).—The alloy contains 0.2—0.5% Cu, up to 0.2% C and Mo (preferably less than 0.1% of each), and other inclusions less than 0.25%.

C. A. KING.

Treatment of alloys. N. B. PILLING, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,671,484, 29.5.28. Appl., 2.9.22).—Iron-silicon alloys (4—8% Si) are mechanically treated at temperatures above 30° and below visible heat. F. G. CROSSE.

Hard-alloy-steel process. C. L. DELACHAUX (U.S.P. 1,671,572, 29.5.28. Appl., 8.12.24. Fr., 8.12.23).—Manganese steel and ordinary carbon steel are welded together by means of an aluminio-thermic mixture containing 18—20% Ni. F. G. CROSSE.

[Welding] alloy. W. F. STODDY (U.S.P. 1,671,384, 29.5.28. Appl., 12.11.25).—Iron is alloyed with 15—35% Cr, 4—16% Mn, and 1—4% Si. F. G. CROSSE.

Cleaning and pickling of metals. G. D. CHAMBERLAIN, Assr. to R. T. VANDERBILT CO., INC. (U.S.P. 1,671,627, 29.5.28. Appl., 19.2.27).—A bath containing sulphuric acid and a small proportion of a thiazole is claimed. F. G. CROSSE.

[Copper-silicon] alloys and method of working and heat-treating the same. M. G. CORSON (B.P. 278,355, 26.9.27. U.S., 4.10.26).—Alloys of copper containing 3.7—6.7% Si are heat-treated at 500—800° to form a solid solution, hot-worked to the desired form, and quenched from above 500°. Particular alloys containing 5% Si and 6.5% Si are treated at temperatures above 700° and 720° respectively. C. A. KING.

Production of metals [zinc] from ores. H. G. S. ANDERSON (U.S.P. 1,669,406, 15.5.28. Appl., 11.9.24).—Zinc sulphide ores are mixed with finely-divided iron and the mixture is heated at such a temperature that the zinc reduced from the ore is volatilised without melting the ferrous sulphide formed in the reaction. A. R. POWELL.

Magnesium alloys for use with pistons for internal-combustion engines. I. G. FARBERIND. A.-G. (B.P. 275,985, 8.8.27. Ger., 13.8.26).—Magnesium alloys containing up to 15% Al and 1—6% Si are claimed. F. G. CROSSE.

Production of aluminium in electric furnaces. V. VERNAY, and SOC. ITAL. ELETTROCHIMICA (B.P. 291,219, 19.5.27).—The metal is produced from aluminium hydroxide obtained by complete decomposition of hydrated aluminium chloride by means of heat. F. G. CROSSE.

[Non-corrosive] aluminium alloys. H. SCHORN, Assee. of R. MÜLLER (B.P. 277,702, 19.9.27. Ger., 18.9.26).—Aluminium is alloyed with 0.05–0.4% of titanium either by direct melting or during the electrolysis of alumina containing titanium compounds or titanous acid in the required proportions. F. G. CROSSE.

Metal alloy. L. R. BRINK, Asst. to BARNHART BROS. & SPINDLER (U.S.P. 1,671,952, 29.5.28. Appl., 7.6.26).—The alloy contains 90% Al, 5% Cu, and 5% Ni, approximately. F. G. CROSSE.

Alloy. M. G. CORSON, Asst. to ELECTRO METALLURGICAL Co. (U.S.P. 1,671,408, 29.5.28. Appl., 13.4.26).—A non-ferrous alloy contains 15–25% Mn, 0.1–3% of an element having the properties of phosphorus and silicon, and the balance copper. F. G. CROSSE.

Welding rod. R. FRANKS, Asst. to OXWELD ACETYLENE Co. (U.S.P. 1,671,417, 29.5.28. Appl., 21.1.26).—An alloy contains 1–12% W, 25–35% Cr, 0.1–1% B, 1–3.5% C, 0.5–3% Si, and sufficient manganese to deoxidise the alloy, the ratio of silicon to manganese being at least 0.9. F. G. CROSSE.

Extraction of tin mixture from lead and tin alloys. M. SPEICHERT (U.S.P. 1,669,580, 15.5.28. Appl., 18.9.26. Ger., 11.10.24).—Scrap bearing metal containing lead, tin, antimony, copper, and other metals is heated until completely liquid, then cooled slowly to just above the m.p. of the eutectic, and the still liquid portion, consisting of a tin-lead alloy with 54.5–55% Sn, up to 3.6% Sb, and up to 0.2% Cu, is separated from the remainder. A. R. POWELL.

Preparation of solder [in flake form]. H. CHAPMAN (B.P. 290,891, 27.9.27).—Molten solder is poured through a sieve into cold water. The flakes so produced are dried and mixed with flux. F. G. CROSSE.

Metal reclaiming process. W. G. NORDLING and A. STEWART, JUN. (U.S.P. 1,669,485, 15.5.28. Appl., 17.3.26).—The metallic mixture is subjected to such a temperature that one constituent is melted; the whole is then agitated and the molten metal removed. The residue is then immersed in a bath of molten flux at a temperature below the m.p. of the residue, but above that of the low-melting constituent, whereby a further quantity of the latter is extracted. A. R. POWELL.

Electrolytic deposition of tin from alkaline solutions. SIEMENS & HALSKE A.-G. (B.P. 290,552, 19.3.28. Ger., 13.10.27. Addn. to B.P. 286,673; B., 1928, 452).—In the recovery of tin by electrolysis a solution of sodium stannate, soluble or insoluble anodes may be used, and an adherent coating of tin may be deposited on a metal cathode, e.g., iron plates. C. A. KING.

Manufacture of rust-proofing material. PARKER RUST-PROOF Co., Assecs. of H. H. WILLARD and M. GREEN (B.P. 270,680, 25.4.27. U.S., 10.5.26).—See U.S.P. 1,660,661; B., 1928, 373.

Recovery of zinc from ores. R. T. D. WILLIAMS and S. W. ROSS, Assrs. to ELECTROLYTIC ZINC Co. of AUSTRALASIA, LTD. (U.S.P. 1,672,016, 5.6.28. Appl., 11.9.26. Austral., 17.9.25).—See B.P. 258,575; B., 1927, 528.

Casting of ingots. (SIR) C. A. PARSONS and H. M. DUNCAN (U.S.P. 1,671,253, 29.5.28. Appl., 6.10.27. U.K., 29.4.26).—See B.P. 278,032; B., 1927, 912.

Treatment of metal surfaces [against corrosion]. W. SMITH, Asst. to EXPANDED METAL Co., LTD. (U.S.P. 1,672,180, 5.6.28. Appl., 3.3.27. U.K., 17.3.26).—See B.P. 272,610; B., 1927, 606.

Production of designs on celluloid, metal, etc. D. A. TUCK, A. FORT, and R. TUCK & SONS, LTD. (B.P. 290,871, 24.8.27).

Blast tuyères for shaft furnaces. GUTEHOFFNUNGSHÜTTE OBERHAUSEN A.-G., A. WAGNER, and E. POHL (B.P. 290,861, 22.7.27).

Ovens (B.P. 289,128).—See I. Amalgamator (U.S.P. 1,669,430).—See XI.

XI.—ELECTROTECHNICS.

Improvement in the technique of setting up standard cells. M. EPPLEY (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 11 pp.).—An improved technique for the preparation of normal cadmium cells is described. The methods are devised with the object of removing any atmospheric oxygen which might be trapped between the mercury and the H-vessel, or air from the inside walls of the latter. Measurements of the *E.M.F.* indicate a high degree of uniformity and values in good agreement with existing standards. H. J. T. ELLINGHAM.

Cathode equilibrium in the cadmium standard cell. M. EPPLEY (Amer. Electrochem. Soc., April, 1928, 53. Advance copy. 13 pp.).—Experiments have been carried out with the object of explaining the irregularities observed by Hulett on rotating the cathode system of the standard cadmium cell. It is concluded that the irregularities are not due to hydrolysis of the mercurous sulphate to a basic sulphate when the sulphuric acid concentration in the electrolyte is greater than about 0.002*M*. The equilibrium in the system containing mercury, mercurous sulphate, hydrated cadmium sulphate, and saturated cadmium sulphate which is 0.002–0.12*M* to sulphuric acid is not the same as that in the same system with mercury omitted. Mercury affects the equilibrium, as shown by *E.M.F.* measurements, but does not appreciably alter the mercury concentration as shown by analysis. Hence a new compound of low solubility is probably formed. Its formation is less rapid the lower the acid concentration. The existence of three forms of mercurous sulphate is indicated by solubility data. One of these should be the basic mercurous salt of Gouy (B., 1900, 683) and Cox (B., 1904, 758). H. J. T. ELLINGHAM.

Tests on paints. BECKER.—See XIII. Ash in sugar. ŠANDERA and ZIMMERMANN.—See XVII.

PATENTS.

Electric furnace. C. H. GAGE, Asst. to L. W. SEARLES (U.S.P. 1,671,026, 22.5.28. Appl., 12.2.26).—Spaced electrodes are arranged at the longitudinal axis of a furnace chamber comprising upper and lower parts, which is substantially elliptical in longitudinal section and circular in cross-section. End electrodes

terminating at the longitudinal axis of the chamber are spaced from the ends of the chamber for a distance equal to the radius of cross-section of the chamber, and a co-acting electrode element is arranged in the bottom of the chamber with its top near the central longitudinal axis of the chamber and with its sides inclined and converging upwards so that the greater part of the bottom of the chamber is uncovered.

J. S. G. THOMAS.

[Filaments for] electric incandescence lamps. GEN. ELECTRIC CO., LTD., ASSEES. OF PATENT-TREUHAND-GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (B.P. 283,848, 15.12.27. Ger., 17.1.27).—The surface of the coiled lamp filament is roughened by sand-blasting, by slow chemical or electrolytic etching (e.g., with a 3% potassium ferricyanide solution), or by deposition of material upon the wire from the gaseous state, e.g., by deposition of tungsten from tungsten hexachloride.

J. S. G. THOMAS.

Treatment of filaments for incandescence lamps etc. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF G. E. INMAN and W. P. ZABEL (B.P. 282,812, 29.12.27. U.S., 31.12.26).—Filaments are cleaned and fired in a container in which they are maintained in definite relation to one another.

J. S. G. THOMAS.

Preparation of a tungsten filament. F. BLAU, F. KOREF, and K. MOERS, ASSRS. to GEN. ELECTRIC CO. (U.S.P. 1,670,292, 22.5.28. Appl., 29.11.24. Ger., 14.2.24).—In a filament that does not readily assume the macro-crystalline form when heated, strains sufficient to form nuclei of crystallisation are produced at several points along the filament, which is then heated to allow macro-crystallisation to set in. H. ROYAL-DAWSON.

Electron device and method of activation. H. C. RENTSCHLER, ASSR. to WESTINGHOUSE LAMP CO. (U.S.P. 1,670,483, 22.5.28. Appl., 4.5.23).—The refractory-metal filament of an electron tube is activated by means of a coating of radium oxide.

H. ROYAL-DAWSON.

Electrical, gas-filled discharge tubes. F. MEYER, H. J. SPANNER, and E. HERFURT (B.P. 275,552, 18.2.27. Ger., 6.8.26).—The gas or vapour pressure in an incandescent cathode rectifier is maintained by means of activated carbon, completely saturated with gas, e.g., neon placed in a container, and, if desired, connected with an electrode.

J. S. G. THOMAS.

Electrical precipitation of small particles from gases. LODGE-COTTRELL, LTD., and L. LODGE (B.P. 290,030, 23.4.27).—The high-tension electrode is hollow and the gases are supplied to its interior through a length of insulating pipe; the gases emerge through a large number of small orifices provided with sharp edges or a number of points to ensure an effective charge being given to the dust particles in the gas currents, which must move at fairly high speed to keep the orifices clear. The high-tension electrode is surrounded by the other (preferably earthed) electrode, which rotates against a fixed scraper on its inner surface, or a moving scraper rotates against the inner surface of a fixed electrode. Several means are suggested for cooling the outer surface of the earthed electrode.

B. M. VENABLES.

Centrifugal amalgamator. R. THAYER (U.S.P. 1,669,430, 15.5.28. Appl., 1.5.22).—The apparatus comprises a hollow revolving cathode, which serves as the outer wall of the centrifugal basket, and an axial pulp intake provided with a metal deflector across its outlet to act also as an anode, so that the centrifugal repulsion of the amalgam and particles of valuable metal is assisted by the current flowing from the anode to the cathode.

A. R. POWELL.

Thermo-electric pyrometers. J. L. ORCHARD and CAMBRIDGE INSTRUMENT CO., LTD. (B.P. 290,493, 14.9.27).—In an indicating thermo-electric pyrometer for use over a definite narrow range of temperature, a bimetallic compensator used to compensate for variations of temperature of the cold junction is arranged also to compensate for temperature changes of the indicator within the temperature range.

J. S. G. THOMAS.

Precipitation of metals on an incandescent body. A. E. VAN ARKEL and J. H. DE BOER, ASSRS. to N. V. PHILIPS' GLOEILAMPENFABR. (U.S.P. 1,671,213, 29.5.28. Appl., 12.3.26. Holl., 14.3.25).—See B.P. 249,067; B., 1927, 195.

Manufacture of carbons for use as anodes of electric cells. COMP. LORRAINE DE CHARBONS, LAMPES, & APPAREILLAGES ELECTRIQUES (B.P. 285,415, 17.3.27. Fr., 16.2.27).

[Portable apparatus for] electrodeposition of metals. J. F. BENNETT and J. HADFIELD (B.P. 290,704, 22.1.27).

Discovering and determining the position, configuration, and nature of ores, solutions of salts, or other bodies. AKTIEBOLAGET ELEKTRISK MALM-LETNING (B.P. 271,871, 23.5.27. Swed., 26.5.26).

[Sheaths for] electrodes of secondary batteries. "HART" ACCUMULATOR CO., LTD., and W. SCHOFIELD (B.P. 291,138, 22.2.27).

Per-compounds (B.P. 290,750).—See VII. Tin. (B.P. 290,552).—See X. **Insulating varnishes etc.** (B.P. 273,756).—See XIII.

XII.—FATS; OILS; WAXES.

Utilisation of the soya bean. II. Y. TAKAYAMA (J. Soc. Chem. Ind. Japan, 1928, 31, 319—322).—The bean was ground to powder after the removal of oil and bran, and digested with sulphuric acid (1—0.025*N*) at 100° for from 1 to 19 hrs. The amounts of dissolved protein and reducing sugar increased as the total amount of acid added increased; the concentration of the acid did not affect the time required for the dissolution.

S. OKA.

Pracaxy and owala kernels and their oils. L. MARGAILLAN, A. DUPUIS, and J. ROSELLO (Ann. Musée Colonial Marseille, 1925, 3, 23—28; Chem. Zentr., 1928, i, 435).—Pracaxy (pracachy) kernels, from *Pentaclethra filamentosa* (Brazil), contain water 10.6, ash 1.4, protein 14.4, fat and resin 48.3, cellulose 4.2, and nitrogen-free extract 21.1%. The yellow, slightly bitter fat has m.p. 28°, n_D^{20} 1.4561, saponif. value 182, acid value 3.6, iodine value 67.3, Reichert-Meissl value 1.2, acetyl value 54.2, resin content

(mol. wt. 470) 0.7%. Owala kernels, from *P. macrophylla* (Congo), contain water 8.5, ash 2.1, protein 25, fat 41, cellulose 5, and nitrogen-free extract 18.4%. The oil has m.p. 24°, f.p. 23–10°, n_D^{20} 1.4682, d_4^{20} 0.902, saponif. value 181, acid value 3.7, iodine value 98.9, Reichert–Meissl value 1.27, acetyl value 0, resin 0.5%. The average mol. wt. of the fatty acids was 309.

A. A. ELDRIDGE.

Jaboty kernel oil. L. MARGAILLAN (Ann. Musée Colonial Marseille, 1925, 3, No. 3, 37–38; Chem. Zentr., 1928, i, 435).—Jaboty kernels (Brazil) contain water 5.7, ash 4, protein 7.9, oil 53.7, cellulose 12.4, nitrogen-free extract 16.3%. The oil has m.p. 45°, f.p. 36°, n_D^{20} 1.4553, saponif. value 228, iodine value 23.1, Reichert–Meissl value 1.3, acetyl value 32, unsaponifiable matter 1.6%, resin 3%. The insoluble fatty acids (94.3%) have f.p. 35.1°, mean mol. wt. 259, and contain liquid acids 25%, stearic acid 3.4%, palmitic acid 43.6%, myristic acid 28%.

A. A. ELDRIDGE.

Properties of "Yamagobo" oil. M. OGURA (J. Soc. Chem. Ind. Japan, 1928, 31, 60).—The oil, obtained from the seed of "yamagobo" (*Phytolacca acinosa*, Roxb.), has d_4^{25} 0.9148, n_D^{20} 1.4713, saponif. value 186.2, iodine value 104.6, and unsaponifiable matter 1–73%. The fatty acids separated from the oil are liquid, have acid value 198.5, iodine value 105.7, and contain 92.3% of liquid acids and 7.7% of solid acids. The fatty acids give 35.9% of a solid bromide, having m.p. 113–114°, which corresponds to that of tetrabromostearic acid.

K. KASHIMA.

Comparative antirachitic value of various marine animal oils and of cod liver oil. L. RANDOIN, E. ANDRÉ, and R. LECOQ (J. Pharm. Chim., 1928, [viii], 7, 529–539).—Rat-feeding experiments indicate that the antirachitic values of the liver oils of *Merluccius merluccius*, L. (hake), and of *Galeus canis*, Rondelet (dogfish), are, respectively, 10 and 5% of that of cod liver oil. The blubber oil of *Macrorhinus leoninus*, L. (sea-elephant), gave negative results. The constants of the oils used are given.

B. FULLMAN.

Reaction of [edible] oils to ultra-violet light. R. MARCILLE (Ann. Falsif., 1928, 21, 189–197).—Certain edible oils give characteristic fluorescence in ultra-violet light. The application of this property to the detection of adulterants in olive oil is discussed.

T. M. A. TUDHOPE.

Occurrence of disperse phases in drying oils. H. EIBNER and A. GRETH (Chem. Umschau, 1928, 35, 97–100; cf. B., 1928, 340, 375).—The experimental basis of Auer's isocolloidal theory of the drying of oils has been reinvestigated by submitting representative specimens to the action of 95%, 47%, and absolute formic acid. Using unoxidised linseed oil, cold-pressed from the seed and preserved in an atmosphere of carbon dioxide, commercial raw linseed oil, or a fresh, light, air-bleached linseed oil, it was impossible to detect the formation of three layers or to isolate a disperse phase. Under the same treatment varnishes and polymerised stand oils separated into three layers, the middle one affording a lower disperse phase insoluble in acetone. The disperse phase had a lower iodine value and a much

more rapid rate of drying than the oily disperse medium. Fresh perilla oil furnished no disperse phase in spite of its rapid rate of drying, whilst fresh wood oil and various polymerised wood oils afforded three layers, and the disperse phase could be isolated in some cases by filtration. There are no grounds for assuming the presence of two phases in freshly-prepared drying oils; the disperse phase makes its appearance only on heating in an amount proportional to the time of heating. In the case of polymerised oils, "drying" is a colloid-chemical phenomenon. Stand oils afford more disperse phase than linseed oil varnishes. The occurrence of a disperse phase in Chinese wood oil as it appears on the European market is due to the fact that it is heated during manufacture in China. Auer's purely colloid-chemical theory of the drying process is too general.

S. COFFEY.

Determination of iodine values [of oils] by Hanus' method. M. WIERNIK (Farben-Ztg., 1928, 33, 1973).—Linseed and Chinese wood oils have been found to give different iodine values by the Hübl–Waller and Hanus methods, the latter usually being much higher. Polymerised oils show bigger deviations than unpolymerised oils.

S. COFFEY.

PATENTS.

Transference of vitamins [from fatty materials]. —S. GRÖNNINGSAETER, and FISCHER-HOLLINSHED Co., Inc. (B.P. 280,212, 2.11.27. U.S., 2.11.26).—The vitamins obtained as an extract on treating a saponified, vitamin-rich, fatty material with an organic solvent may be transferred directly to a vitamin-deficient fatty material by adding the foregoing mixture to the substance to be enriched and then adding a diluent. Thus, if the solvent be alcohol, simple addition of water to the resulting mixture is sufficient to cause the separation of the required enriched fatty oil from the alcohol and the soap of the original fatty material. It is desirable to carry out these operations at moderate temperatures.

E. HOLMES.

Floor, boot, and like polishes. Benzene soaps. I. G. FARBERIND. A.-G. (B.P. 261,039 and 287,114, 8.11.26. Ger., 9.11.25).—(A) Hydrogenation benzene or its cracked, polymerisation, dehydrogenation, or demethylation products are suitable for incorporation with floor and boot polishes; they have enhanced miscibility with other organic solvents and are less inflammable. (B) Benzene soaps made with hydrogenation benzene or its products are claimed to have a good solvent power for dirt and impurities.

E. HOLMES.

Treatment of crude cottonseed oil. D. McNICOLL, Assr. to BRIT. OIL & CAKE MILLS, LTD. (U.S.P. 1,671,834, 29.5.28. Appl., 12.1.25. U.K., 6.2.24).—See B.P. 232,361; B., 1925, 557.

Expressing liquids from solids (B.P. 289,954).—See I. Phosphatides (U.S.P. 1,667,767).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

"Wetting-out agents" for the paint industry and their investigation. A. NOLL (Farben-Ztg., 1928, 33, 2293–2299).—A general dissertation on the chemical nature of wetting-out and emulsifying agents used in

water-paint manufacture (chiefly sodium salts of complex organic sulphonic acids). Details of the composition, properties, purification, and fluorescence characteristics of various commercial products of this type, *e.g.*, Nekal, Leonil, Betan, Majamin, Algosol, etc., are given, and the stability of emulsions formed by the aid of various combinations of these is tabulated. S. S. WOOLF.

Ultra-violet light and its use in accelerated weathering tests on paints. E. BECKER (Farben-Ztg., 1928, 33, 2232—2234).—In a general theoretical discussion on the nature of electromagnetic waves it is shown that the use of the light emitted by the mercury-vapour lamp to represent the action of sunlight in accelerated weathering tests on paints etc. must give erroneous results. The mercury vapour lamp emits short-wave radiation down to λ 185 $\mu\mu$, whilst the radiation from the sun contains no waves shorter than 300 $\mu\mu$. It follows, therefore, that the greater energy quanta liberated by the former source induce reactions that cannot occur under the influence of sunlight. It is suggested that filtration of the mercury-vapour emission through suitable glass filters in order to eliminate the wave region not present in sunlight would render the artificial source applicable to accelerated weathering tests. S. S. WOOLF.

Colloid-chemical problems [in paint industry]. A. V. BLOM (Farben-Ztg., 1928, 33, 1969—1972).—A lecture dealing with general aspects of wetting power and interfacial tension applied to pigments and paints. S. COFFEY.

Cellulose acetate. ATSUKI and others. **Cellulose ester solvents.** WOLFF.—See V. **Disperse phase in drying oils.** EIBNER and GRETH.—See X.

PATENTS.

Water paints, wood finishes, and the like. W. W. TAYLOR (B.P. 290,700, 20.1.27).—A water-resisting emulsion is made by adding a drying oil having free fatty acid to an aqueous solution of a primary or single alkaline alginate or a mixture of two or more alginates, with or without pigment. B. FULLMAN.

Materials for use in painting or varnishing [or as impregnating agents]. G. KING and R. THRELFALL (B.P. 290,717, 16.2. and 15.3.27).—Silicic acid esters are treated with a solvent, *e.g.*, alcohol or acetone, containing in solution or suspension water in quantity not exceeding that required for hydrolysis, a smaller quantity being used if desired. The product, after expulsion of hydrogen chloride by treatment at about 100° with a current of air, may be mixed with other material, *e.g.*, a pigment or a mixture of such with a solvent, *e.g.*, cyclohexanol, which does not interfere with the usual setting time of the silica solution. L. A. COLES.

Painting and preserving stone, wood, metal, etc. in buildings and sculptures. H. SCHMID (B.P. 290,331, 7.2.27).—A mixture of a neutral wax, *e.g.*, beeswax, with colouring material is applied, molten or in solution, and is then heated. A further coat of an oil colour mixed with wax is given, and, finally, a coating of wax. L. A. COLES.

Manufacture of a red oxide of iron, suitable for use as a pigment. T. STORER and C. J. A. TAYLOR (B.P. 290,421, 9.4.27).—A solution containing a ferrous salt, *e.g.*, galvanisers' or enamellers' waste liquor neutralised with scrap iron, is heated under pressure with manganese dioxide or other suitable non-gaseous oxidising agents, with or without the addition of lime or calcium carbonate, and the precipitate is collected, washed, and dried. Alternatively, the reaction is allowed to proceed at the ordinary temperature, and the precipitate, after removal of the supernatant liquor, is heated under pressure. L. A. COLES.

Manufacture of white lead. HOLZVERKOHLUNGS-IND. A.-G., and J. SEIB (B.P. 290,844, 9.6.27).—Basic lead carbonate containing 13.6% CO₂, obtained by the addition of sodium carbonate to a solution containing 0.25 mol. of lead oxide per mol. of lead acetate, is heated under pressure, with or without removal of the lead acetate lye, until the product contains about 11.4—11.5% CO₂. L. A. COLES.

Manufacture of lacs, varnishes, and enamels suitable for insulating purposes. L. LEVY (B.P. 273,756, 1.7.27. Fr., 3.7.26).—"Thiolite" (cf. B.P. 184,164; B., 1923, 560 A), prepared at a low temperature and under conditions favourable for the formation of a minimum of free sulphur, is finely ground and washed, after which it is dissolved in a suitable solvent. The resultant varnish is dried, after application, at 110—160°. L. A. COLES.

Manufacture of varnishes, lacquers, and the like. L. MCG. FRASER and W. S. SMITH (B.P. 290,707, 8.2.27).—Apparatus is described which effectively encloses the components during the operations, and mixing etc. is carried out in an entirely enclosed, rotatable drum with internal vanes, with means for filling and emptying it by pressure. Vapours arising are condensed and returned by reflux condenser or otherwise. B. FULLMAN.

Oil varnishes containing resins. Cellulose ester lacquers. I. G. FARBENIND. A.-G. (B.P. 287,115—6, 8.11.26. Ger., 9.11.25).—(A) Benzines obtained by destructive hydrogenation (*e.g.*, from lignite tar or crude American mineral oil) or the products obtained from them by cracking, polymerisation, dehydrogenation, or demethylation, are used in making oil varnishes containing resins. For example, 10 pts. of a linseed oil-kauri gum varnish (oil:gum = 1:2, 1:1, or 2:1) are mixed with 10 pts. of benzine. (B) Similar products are used as diluents in making cellulose ester lacquers. B. FULLMAN.

Preparation of surfaces for the reception of cellulose lacquers. I. G. FARBENIND. A.-G. (B.P. 281,310, 24.11.27. Ger., 24.11.26).—The surfaces are coated with an intermediate layer consisting of a protein, a resin, and a softening agent (*e.g.*, casein glue, shellac, and camphor in ammoniacal aqueous alcohol). B. FULLMAN.

Production of porous artificial compositions. KUNSTHARZFABR., and Dr. F. POLLAK GES.M.B.H. (B.P. 261,409, 13.11.26. Austr., 14.11.25).—The gelatinous product obtained by the condensation of carbamide

and formaldehyde is allowed to harden without removal of the water, the bulk of the water is then removed below 30°, and the final hardening and drying is effected at a high temperature. The appearance of the product depends on the size of the pores, which is of the order of 0.4–4.0 μ , and is varied by the degree of dilution during condensation. L. A. COLES.

Manufacture of condensation products of urea and formaldehyde. I. G. FARBENIND. A.-G. (B.P. 261,029, 6.11.26. Ger., 7.11.25).—Dimethylolurea dissolved or suspended in an organic solvent which may consist of or contain phenols, but is free from water, is heated in the presence of an acid condensing agent, *e.g.*, alcoholic hydrogen chloride solution. L. A. COLES.

Manufacture of condensation products of urea and its derivatives and formaldehyde. I. G. FARBENIND. A.-G. (B.P. 264,466, 10.12.26. Ger., 12.1.26. Addn. to B. P. 259,950; B., 1928, 165).—The condensation products are hardened by exposure to acids (*e.g.*, formic, hydrofluoric) or acid anhydrides, gaseous or dissolved, and, if desired, under high or low pressure. B. FULLMAN.

Production of resinous media. R. ARNOT (B.P. 290,327, 15.1., 26.3., and 2.5.27).—An aldehyde (formaldehyde) is condensed in one or in two stages with an organic amino-compound (carbamide, calcium cyanamide, aromatic or aliphatic amines) and an organic acid or salt or a phenol or naphthol. Acid or alkaline catalysts may be used. Carbamide, formaldehyde, and salicylic acid give according to proportions used clear oils, white solids, or gums. With sodium salicylate adhesives are obtained. The amount of formaldehyde necessary for condensing with a mixture of phenoxides and carbamide is much less than for carbamide alone. Formaldehyde condensed with hydroxy-acids and then with carbamide gives glassy products, and very tough resins are obtained when arylamines are a constituent. Succinic or cinnamic acid with formaldehyde and carbamide gives transparent, glass-hard products. C. HOLLINS.

Production of unsaturated organic compounds [artificial resins]. E. FREUND, ASSP. to CHEM. FABR. AUF ACTIEN VORM. E. SCHERING (U.S.P. 1,672,378, 5.6.28. Appl., 1.9.22. Ger., 22.9.21).—See G.P. 417,667; B., 1926, 100.

Vat dyes (B.P. 273,247).—See IV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Vulcanisation of rubber. N. BACON (J. Physical Chem., 1928, 32, 801–828).—Experiments are described such as the heating of a mixture of purified vulcanite (shavings or solution, *e.g.*, in cresol or aniline) and raw rubber (solid or solution), the fractional extraction of vulcanised rubber or of a mixture of this and unvulcanised rubber by benzene or xylene, and also the heating of rubber with an organic accelerator and sulphur in butyl alcohol. The results confirm, in its essential features, the early theory of Weber which regards the union between rubber and sulphur in vulcanisation as a chemical one. From the homogeneity of vulcanisates

containing as little as 0.1% S, the view is expressed that the mol. wt. of caoutchouc is at least 32,000.

D. F. TWISS.

Fineness and reinforcing power of rubber fillers. W. ESCH (Gummi-Ztg., 1928, 42, 1921).—The degree of fineness of a compounding ingredient as measured by dividing the weight of 1 c.c. of the closely packed powder by the true sp. gr. falls approximately in the reverse order to its reinforcing power in rubber. The indication is of greater significance in the comparison of fillers of similar chemical composition, the relationship being complicated, apparently, by the chemical action of the natural resin acids on some types of filler. D. F. TWISS.

Lamellar packing of caoutchouc. M. KRÖGER (Kolloid-Z., 1928, 45, 46–52).—A description is given of experiments on the properties of caoutchouc under lamellar deformation and under stretching. The course of the increase in potential energy, the capacity for such increase, the optical and thermal effects, together with the changes in density and electrical conductivity were investigated for different degrees of deformation and of vulcanisation and at different temperatures: the effect of added substances was also examined. The microscopical appearance of the surface was examined, and an apparatus for its measurement is described. E. S. HEDGES.

PATENTS.

Manufacture of rubber and similar vegetable resins. DUNLOP RUBBER Co., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 290,313, 13.11.26 and 10.3.27).—Compounding ingredients such as fillers, reinforcing agents, softeners, and agents and accelerators of vulcanisation, previously dispersed in water if desired, are mixed with latex in the presence of one or more stabilisers, *e.g.*, alkali, glue, gelatin, or casein, and the mixtures are concentrated by evaporation while being continuously subjected to dispersive agitation. The introduction of vulcanising agents may be made during the concentration process. Smooth stable creams containing up to 80% of total solids may be thus obtained; their stability and fluidity may be increased by the addition of ammonia; they are especially suitable for the manufacture of articles by spreading, dipping, or deposition processes. D. F. TWISS.

Production of rubber mixings from latex and filling materials. K.D.P., LTD. (B.P. 277,374, 12.9.27. Ger., 10.9.26).—Latex, preferably concentrated to a paste, is mixed with filling materials, especially lamp black, while constantly forming new surfaces over which the filling materials can be uniformly distributed; the mixture is simultaneously dried. Two heated rollers, one of which is fluted, constitute a convenient device for the operation. It is possible in this way to obtain a mixture containing rubber with twice its weight of gas black. D. F. TWISS.

Manufacture of cellular rubber and elastic tyres therefrom. F. RINGER (B.P. 290,447, 26.5.27).—Cellular rubber is produced by intimately incorporating carbonates or bicarbonates of heavy metals, *e.g.*, white lead, in a plastic rubber mass and vulcanising

by heat, whereby carbon dioxide is concurrently liberated giving rise to closed cells. By effecting vulcanisation of the mass when occupying part of the internal space of a tube of rubber and/or fabric, the mass swells so as to fill the available space. The unvulcanised mass also may be introduced inside a mould lined with an ordinary rubber mixing; on vulcanisation a rubber tyre is obtained with a cellular interior encased in a solid rubber shell.

D. F. TWISS.

Preservation of [rubber] latex. I. G. FARBENIND. A.-G. (B.P. 289,022, 30.3.28. Ger., 20.4.27).—Coagulation of latex is prevented by the addition of water-soluble, neutral, or alkaline salts of ester acids, *e.g.*, diarylphosphoric acids or methylsulphuric acid; sodium dicresyl phosphate, ammonium diphenyl phosphate, and potassium methyl sulphate are mentioned.

D. F. TWISS.

Preparation of age-resisting rubber compositions. E. I. DU PONT DE NEMOURS & Co., Assces. of H. W. ELLEY (B.P. 276,968, 26.7.27. U.S., 31.8.26).—Secondary aromatic amines, preferably diarylamines, containing substituted or unsubstituted phenyl or naphthyl groups, which are substantially without accelerative action on vulcanisation, are used to impart age-resisting qualities to rubber. Phenyl- and tolyl- α -naphthylamine and diphenyl-, ditolyl- and dixylylamine are mentioned. They are easily dispersed in rubber, and do not lead to "spotting" in the rubber when stored.

D. F. TWISS.

Vulcanisation of rubber. C. O. NORTH and C. W. CHRISTENSEN, Assrs. to RUBBER SERVICE LABORATORIES CO. (U.S.P. 1,670,312, 22.5.28. Appl., 19.5.24).—Vulcanisation is effected with the aid of the reaction product of aniline and crotonaldehyde.

D. F. TWISS.

Manufacture of rubber-like products. W. O. HERRMANN and W. HAEHNEL, Assrs. to CONSORT. F. ELEKTROCHEM. IND. (U.S.P. 1,672,157, 5.6.28. Appl., 8.11.26. Ger., 23.11.25).—See B.P. 261,748; B., 1928, 132.

Concentration of caoutchouc and the like latices. A. PETERSON (U.S.P. 1,670,900, 22.5.25. Appl., 12.11.25. Ger., 14.11.24).—See B.P. 243,016; B., 1926, 956.

Aldehyde-amine condensation products (B.P. 265,931).—See III.

XV.—LEATHER; GLUE.

Capacity of chrome-tanned leather for absorbing sulphonated and non-sulphonated fats. E. MEZEY (Collegium, 1928, 209—213).—Samples of chrome-tanned leather were fat-liquored with mixtures of sulphonated neatsfoot and untreated neatsfoot oil, sulphonated neatsfoot and mineral oils, sulphonated fish oil and fish oil, and sulphonated fish oil and mineral oil, respectively. The total amount of fat used was constant, and the dry leather was analysed. It is shown that the amount of fat absorbed increases with increase in the proportion of non-sulphonated oil to a maximum beyond which the amount of sulphonated oil is insufficient to emulsify the other oil. The efficacy of a fat-liquor depends not only on the amount but on its composition. The non-sulphonated oil is absorbed

much more readily by chrome leather than is the sulphonated oil.

D. WOODROFFE.

Migration of the masked sulphate groups in chrome alum liquors. W. SCHINDLER and K. KLANFER (Collegium, 1928, 97—107).—Boiled green solutions of chrome alum slowly give up a part of their masked sulphate groups. Additions of sodium carbonate lowered the amount of such groups. The latter depends to a great extent also on the age of the solution before it is made basic. A 43% basic liquor is much more labile than are less basic liquors. This is particularly so with liquors which have been prepared from fresh green chrome alum solutions, an important point in connexion with their tanning properties (*cf.* Stiasny, B., 1925, 560; Grasser, B., 1921, 709 A). Such changes can be explained by the size of the particles and the degree of "verolung." Dilution does not affect the labile 43% basic liquor. There were occasional increases in the amount of sulphate groups in the chromium complexes, especially with a liquor of 28% basicity, due, possibly, to the decomposition of the carbonato-compounds first formed; in other cases it was caused by variations in temperature. The whole of the sulphate groups in the chromium complex could not be removed by increasing the basicity, even up to 60%. In a few cases on ageing a violet solution there was a migration of sulphate groups into the complex. Chromium chloride liquors behaved differently (*cf.* Stiasny and Balányi, B., 1927, 393). An addition of potassium oxalate removed all the sulphate groups from the chromium complex. Neutral salts and neutral organic compounds are in part very effective in this respect. In tanning pelt there is always an appreciable diminution in the amount of sulphate groups in the chromium complexes in the chrome liquor. Different amounts of sulphate groups in the chromium complex were found in commercial chrome liquors of similar analytical composition. There were roughly two groups, one containing 0.6 mol. of masked (SO_4) per atom of chromium, and the other 0.8 mol. D. WOODROFFE.

Properties of leather. Retan sole leather. A. C. ORTHMANN (J. Amer. Leather Chem. Assoc., 1928, 23, 184—187).—The water-resisting properties of samples of chrome sole leather, which had been retanned with vegetable tannin, have been determined by the water-absorption method and by making and wearing a sandal. The latter is used on a concrete floor covered with water, and the time noted for the feet to become wet. There was no connexion between the water absorbed in $\frac{1}{2}$ hr. or 3 hrs. and the wearing test. Eight leathers advertised as water-resisting wet the feet in 8—43 min. when tested. The content of fatty matter varied from 2% to 21%, but there was no direct relationship between it and the water-resisting properties. D. WOODROFFE.

Analysis of synthetic tannins. Technical condensation products of aromatic sulphonic acids. I. BERKMANN and A. KIPRIANOV (Collegium, 1928, 177—197).—Total sulphur in the dry synthetic tannin is determined by fusion with sodium carbonate followed by treatment with bromine and precipitation with barium chloride in the usual way. The ash and sulphur present in the product obtained by neutralising the synthetic tannin are also determined. Losses due to

reduction of sodium sulphate to sulphide and volatilisation of the ash are negligible. The results are calculated to SO_3 in the dry product. If A is the total SO_3 in the original product and a that in the ash, x the SO_3 in sulphonic groups and y that as sulphate, then $x = 2(A - a)$ and $y = 2a - A$. Dennstedt's method is also applicable, but is more complicated. The value of a can also be derived by adding the percentage of the ash of the original dry product to the acidity of the dry synthetic tannin calculated as SO_3 . The acidity is determined by titration to p_H 7—7.5. When sodium chloride is present in the synthetic tannin, a sample is moistened with sulphuric acid and ashed. To the value obtained is added the acidity of the synthetic tannin. The total chlorine is then determined, and thus the sodium chloride, and expressed as sodium sulphate. This figure is deducted from the sum of the other two determinations and gives a . The purely organic substances of the type $\text{CH}_2(\text{RH})_2$ can be obtained by deducting the percentage of mineral salts from that of the dry neutralised tannin, which gives the pure sulphonated salt, from which the hydrocarbon radical can be calculated. The degree of sulphonation is given by the ratio of combined SO_3 to organic matter, or is better expressed as the average mol. wt. of the sulphonic acid present in the synthetic tannin. The composition of the various commercial products is discussed.

D. WOODROFFE.

Biochemistry of soaking. I. Changes occurring within the skin. E. R. THEIS and E. L. McMILLEN (J. Amer. Leather Chem. Assoc., 1928, 23, 226—233).—Portions of salted hide were soaked for from 0 to 120 hrs. at 25° in water, then surface-washed, digested for 24 hrs. with 20% hydrochloric acid, and the excess acid removed, the residue being taken up in ammonia-free water and separated into the four nitrogen groups (cf. Osborne and Harris, B., 1903, 655). During soaking the amino-nitrogen remains constant, the basic nitrogen decreases greatly during the first 24 hrs. and then only slowly afterwards, and the non-basic nitrogen increases greatly corresponding to the decrease of the basic nitrogen. Analysis of the soaked hide pieces by Van Slyke's method shows that the amount of arginine, lysine, histidine, and imino-nitrogen all decrease, whereas the amino-nitrogen increases correspondingly. It is suggested that the basic amino-acids, particularly arginine, are easily converted by enzymes and bacteria into simpler acids, with the subsequent production of carbamide, which, with the amines, is soluble in the water used for soaking.

D. WOODROFFE.

Determination of insoluble [matter] in tanning extracts. A. TURNBULL (J. Amer. Leather Chem. Assoc., 1928, 23, 224—225).—A method depending on the formation of a suitable filtering medium is described, in which a Schott filter marked "17 G with porous septum No. 1" is fitted into a filter flask and the whole evacuated to a pressure of 15 in. 1 g. of kaolin is then added to 75 c.c. of the tan liquor, stirred, and poured on to the filter, stirring being continued so that the glass rod used gently rubs the whole of the upper surface of the porous septum as the liquor filters through. The liquor is then returned and the process repeated.

D. WOODROFFE.

Measurement of colour of leather with the Pulfrich photometer. F. LÖWE (Collegium, 1928, 197—203).—Applications of this instrument to various samples of leather are described, the particular colour field, percentage of full colour, black, and white, being reported.

D. WOODROFFE.

PATENTS.

Tanning of hides. M. M. MERRITT, Assrs. to UNITED SHOE MACHINERY CORP. (U.S.P. 1,668,652, 8.5.28. Appl., 1.4.22).—Hides are suspended in tanks through which the tan liquor is flowing continuously past them at the rate of over 100 gals./min. The liquor enters the tank at a point below the surface of the liquor near one end and is withdrawn at the other end. A mechanism is arranged to lift the hides into the atmosphere, allow some of the water in the liquor on the hides to evaporate, and then lower them again into the tan liquors.

D. WOODROFFE.

Production of decorative leather from fish skins. S. STRANSKY (B.P. 287,918, 29.3.28. Austr., 29.3.27).—The skins are limed, then two pieces of equal size are fastened together, the inside to the inside, so that they can be readily separated later, and the skins are tanned.

D. WOODROFFE.

Humidification of materials (B.P. 264,538).—See I.

XVI.—AGRICULTURE.

Relation between concentration of mineral elements in a culture medium and absorption and utilisation of those elements by plants. F. W. PARKER and W. H. PIERRE (Soil Sci., 1928, 25, 337—343).—A series of solution culture experiments was set up to determine the minimum concentration necessary for the full growth of maize and soya beans. The values so obtained were compared with the concentrations of these radicals found in displaced soil solutions. The indications are that the soil solution normally contains enough potassium for the complete nutrition of plants, provided that the concentration is maintained during the growing season. The phosphate concentration of the soil solution in many soils is too low, although satisfactory yields of maize are obtained on these soils. This would indicate that root-soil contact is necessary for the phosphate nutrition of plants.

H. J. G. HINES.

Crop-producing power of limited quantities of "essential" plant nutrient. C. HARTMAN, JUN., and W. L. POWERS (Soil Sci., 1928, 25, 371—377; cf. Parker and Pierre, preceding).—Lucerne and wheat were grown in series of water cultures wherein the concentrations of nitrogen, calcium, magnesium, potassium, phosphorus, and sulphur, respectively, varied from nil to a more than adequate amount. It is intended in a further paper to use the results so obtained in comparison with corresponding values for soil solutions in an attempt to discover the minimum and optimum values for plant growth.

H. J. G. HINES.

Relation of concentration of calcium ion required by lucerne to amount present in soil solution. H. W. E. LARSON (Soil Sci., 1928, 35, 399—408; cf. Hartman and Powers, preceding).—Water cultures demonstrated that the minimum concentration of calcium necessary for the growth of lucerne was about 16 p.p.m.

and that the most economical concentration was 32 p.p.m. Determinations of the water-soluble calcium in soils showed that the concentration often fell short of this standard, and that the application of limestone and sulphur remedied this deficiency. Calcium is needed not only to amend the soil reaction, but also as an essential plant nutrient.

H. J. G. HINES.

Influence of soil, season, and manuring on quality and growth of barley, 1926. V. E. J. RUSSELL (J. Inst. Brew., 1928, 34, 307—320; cf. B., 1927, 310).—The year 1926 was abnormal, particularly in its lack of sunshine, and the effect of nitrogenous manure was not quite the same as in the previous four years. At about half the farms the nitrogenous fertiliser gave no increase of crop and in some cases had a harmful effect on the yield. It tended to lower the valuation of the barley and the malt, and to raise the percentage of nitrogen in the barley and lower the 1000-corn weight. The diastatic power of the malts was higher where the barley had received ammonium sulphate, and it was indicated that the diastatic power increased when the nitrogen content of the grain increased. The percentage of nitrogen in the barley did not differ much from those of the previous years, and appeared to be affected more by the soil than by the season or manuring, provided the latter was not too heavy. When the rates of ammonium sulphate were increased there was no increase in barley nitrogen or decrease in the valuation until the amount of manure exceeded 1 cwt. per acre. As in previous years, ammonium chloride proved rather better than the sulphate, since it brought the barley into ear sooner and so made possible a longer ripening period.

C. RANKEN.

Stimulation, toxicity, and antagonism of calcium nitrate and manganese chloride as indicated by growth of wheat roots. L. V. BARTON and S. F. TRELEASE (Bull. Torrey Bot. Club, 1927, 54, 559—577).—Manganous chloride is a stimulant in 0.00001M-solution, and toxic at higher concentrations; calcium nitrate is without effect below 0.00001M, and is toxic above that concentration. Mutual antagonism in mixtures was studied.

CHEMICAL ABSTRACTS.

Comparison of the toxicity and diffusion in a column of grain of chloropicrin, carbon disulphide, and carbon tetrachloride. A. L. STRAND (Minn. Agric. Exp. Sta. Tech. Bull., 1927, 49, 1—59).—A study of relative toxicity to the flour beetle, *Tribolium confusum*, Duv., in relation to concentration, time, and temperature.

CHEMICAL ABSTRACTS.

Susceptibility of wheat to mildew as influenced by salt nutrition. S. F. and H. M. TRELEASE (Bull. Torrey Bot. Club, 1928, 55, 41—68).—Low susceptibility of wheat plants to *Erysiphe graminis* is associated with a high content of potassium dihydrogen phosphate and a low content of calcium nitrate in the culture solution, and *vice versa*. Host vigour and susceptibility are not closely related.

CHEMICAL ABSTRACTS.

Growth of seedlings of conifers in crude humus. I. Transformation of nitrogen in crude humus. H. HESSELMANN. II. Formation of mycorrhiza in connexion with seedlings of *Pinus sylvestris* in different kinds of crude humus. E. MELIN (Rep.

Swedish Inst. Exp. Forestry, 1927, 23, 337—432, 433—494; Proc. Internat. Soc. Soil Sci., 1928, 3, 138—139).—I. The growth of conifer seedlings in sand containing crude humus showed the latter to contain nitrogen in a form capable of rapid conversion into nitrate on exposure. Artificial inoculation with the requisite organisms was important. Crude humus from birch forests proved better than that from pines. There was a very definite connexion between the rate of nitrification of the crude humus, the development of seedlings, and the growth of associated mycorrhiza. The last-named varied with the nature of the humus.

II. The general parallelism between the growth of pine seedlings in humus and the production of mycorrhiza is a direct consequence of the nitrification of the humus, and the rate at which this occurs tends to regulate the symbiotic relationship. A. G. POLLARD.

Calcium ion and root-growth. W. MEWIUS (Jahrb. wiss. Botanik, 1927, 66, 183; Bied. Zentr., 1928, 57, 112—114). O. LOEW (Forts. Landw., 1927, 2; *ibid.*).—The classification of plants according to the effect of calcium salts on their growth is considered in view of the results of treatment with potassium chloride and magnesium sulphate as influenced by reaction and temperature. The theory of the "lime factor" (Loew) is not fully supported by the author's results, which accord more closely with the Hanstein-Cranner conception of the effect of the calcium ion in reducing the permeability of the protoplasm. Other injurious effects follow this action. The stability of protoplasmic permeability is specific to plants.

Loew observes that the death of cells brought about by treatment with salts which precipitate calcium can be watched microscopically, and asserts that the cell nucleus contains important calcium compounds, and when these are decomposed by precipitation the cell must die. This view does not conflict with the fact that potassium carbonate and sodium orthophosphate are less active in destroying protoplasm than are potassium oxalate, sodium fluoride, and sodium pyrophosphate.

A. G. POLLARD.

Action of soil acids on cultivated plants. H. FORKERT (Diss., Hamburg, 1925; Bied. Zentr., 1928, 57, 108—110).—The exchange acidity plays a prominent part in the effect of soil acids on plant growth. In water cultures, nutrient solutions having a p_H value below 5 are injurious. In ranges of less acidity crop increases are obtained. Corresponding effects are observed in soils. Among plants examined in the seedling stage, oats are the most sensitive to acidity, followed in order by barley, rye, and wheat. Peas, flax, and sugar beet are but slightly affected by soil acids. Among cereals, acidity brings little change in straw yields, and the relative grain yield is affected only when the total acidity is above 10 c.c. No morphological differences are noted.

A. G. POLLARD.

Effect of potassium salts on availability of nitrogen in ammonium sulphate. W. B. MACK and D. E. HALEY (Soil Sci., 1928, 25, 333—336).—Potassium chloride appeared to decrease the availability of the nitrogen in ammonium sulphate as judged by the crop yields of a ten-year manurial experiment on crops in

rotation. Laboratory experiments showed that potassium salts had no consistent inhibitory effect on nitrification, and that their adverse effect could not be ascribed to this cause.

H. J. G. HINES.

Technique of pot experiments [with plants]. K. MAIWALD (Landw. Versuchs-Stat., 1928, 107, 342—363).—A review of the whole technique wherein several sources of minor errors are revealed. The Wiley mill is recommended for preparing crops for analysis.

H. J. G. HINES.

Sodium hydroxide rather than sodium carbonate the source of alkalinity in black alkali soils. J. F. BREAZEALE and W. T. McGEORGE (Arizona Agric. Exp. Stat. Tech. Bull., 1927, [13], 307—334).—There is an apparent decrease in the hydroxyl and hydrogen carbonate ions (expressed in terms of dry soil) as the ratio of extracting water to soil is decreased. The true soil solution in most black alkali soils contains few or no hydroxyl ions, no sodium carbonate, and usually very little sodium hydrogen carbonate. Electrometric or colorimetric titration of the soil solution indicates the nature of the components producing the alkaline reaction. The alkalinity of sodium hydroxide produced by the hydrolysis of sodium zeolite is usually mistaken for that of sodium carbonate. In the reclamation of black alkali soils by leaching there is a gradual interaction between sodium zeolite and calcium carbonate. The presence of a common ion, *e.g.*, as sodium chloride, depresses the ionisation of sodium zeolite, and prevents the appearance of hydroxyl ions. If a black alkali soil contains excessive amounts of sodium salts, application of gypsum has little or no effect until the sodium salts are removed by leaching. Free carbon dioxide does not exist in a black alkali soil solution or atmosphere.

CHEMICAL ABSTRACTS.

Toxicity of salines that occur in black alkali soils. J. F. BREAZEALE (Arizona Agric. Exp. Stat. Tech. Bull., 1927, [14], 337—357).—The presence of finely-divided insoluble substances does not affect the toxicity of alkali salts in solution. The endurance limit of a plant for an alkali should be based on the composition of the soil solution at the wilting-point. The toxicity of alkali salts is a function of both molecules and ions. Colloids do not affect the toxicity of sodium carbonate, which does not exist in toxic concentrations in ordinary black alkali soils. Both sodium hydroxide and sodium carbonate act as stimulants to plants in all concentrations usually present in the soil solution of such soils. The infertility of black alkali soils is due to their not readily taking up water, not draining rapidly, and to the fact that they often contain toxic amounts of sodium chloride and sulphate.

CHEMICAL ABSTRACTS.

Aluminium hydroxide in alkaline soils and its effect on permeability. W. T. McGEORGE, J. F. BREAZEALE, and P. S. BURGESS (Arizona Agric. Exp. Stat. Tech. Bull., 1927, [12], 1257—1305).—The appreciable amounts of water-soluble aluminates present in black alkali soils result from the solvent action of alkali hydroxide; when the alkalinity is lowered, *e.g.*, by leaching, colloidal aluminium hydroxide is precipitated, greatly reducing the capillary movement of water and the permeability of the soil for water. For

the reclamation of alkali soils, intermittent leaching and drying, or leaching during the summer when the water is warmer, is advocated.

CHEMICAL ABSTRACTS.

Replaceable bases in some soils from arid and humid regions. M. D. THOMAS (Soil Sci., 1928, 25, 379—392).—Eight clay soils and a natural mineral colloid similar to bentonite were treated so as to exchange their bases for a single base. They were then washed until soluble salts were removed as indicated by the tendency of the soil to disperse. Successive water extracts were then made and analysed, and were compared with water extracts of the untreated soil. The alkali soils showed marked differences in behaviour from the soils from humid regions. These were in the main due to the presence of soluble silicates and the ease with which sodium came into solution after the removal of soluble salts.

H. J. G. HINES.

Exchangeable bases in soil. V. NOVÁK and B. MALÁČ (Bull. Czechoslov. Acad. Agric., 1927, No. 5; Proc. Internat. Soc. Soil Sci., 1927—8, 3, 73—74).—*Carbonate free soils.* The methods of Hissink, Gedroiz, and Kelley for determining exchangeable bases in soils give comparable results. By the Kelley method the sum of the individual bases agrees with the amount of ammonia absorbed from ammonium chloride. In podsol profiles iron and aluminium must be included in the calculation of exchangeable bases. The Gedroiz (0.05N-hydrochloric acid) method in many cases involves some decomposition of the absorbing complex, and in neutral soils the liberation of detectable amounts of iron and aluminium. During the operation of all methods (Hissink not examined) considerable amounts of silica may be rendered soluble. In degraded tschernosem profiles, where the A_1 and A_1' horizons are carbonate-free and the C horizon contains much chalk, only sodium, potassium, magnesium, and calcium were found among the absorbed bases. Iron and aluminium only appeared when the Gedroiz method was adopted. Calcium was usually present in greatest amounts, and sodium and potassium only to a small extent. Podsol profiles with acid reaction differ from tschernosem in having only a small content of total exchangeable bases and by the presence among these of iron and aluminium. In horizons A and A_1 the sum of replaceable iron and aluminium represents 60—70% of the total replaceable bases, whereas in horizons B and C_1 calcium and magnesium are predominant, and the iron and aluminium content falls to 5—9%.

With soils containing carbonates the Hissink and Kelley methods diverge considerably, the former giving values averaging twice those of the latter. Bobko-Askinas's method agrees with Hissink's. Gedroiz's method using 0.05N-ammonium chloride yields irregular results. Using N-ammonium chloride the Gedroiz method gives values double those by Hissink's. Generally speaking, for carbonate-free soils, the Gedroiz simplified method (0.05N-ammonium chloride) is recommended for speed and ease of working. For determining total bases without individual identification, Kelley's method is better. None of the processes examined gives a reliable and satisfactory result for soils containing carbonate.

A. G. POLLARD.

(To be concluded on July 27.)

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 3, 1928.

I.—GENERAL; PLANT; MACHINERY.

References to colloid technology. II. Plastic substances. J. OBRIST (Kolloid-Z., 1928, 45, 82—92).—A review of the literature on plastic substances under the following heads: colloid nature and plasticity, the plasticising process, cellulose products, synthetic resins, proteinoplasts.

E. S. HEDGES.

PATENTS.

Calcining furnaces. A. J. STEPHENS. FROM ZAHN & Co., G.M.B.H. (B.P. 290,837, 2.6.27).—An annular, mechanical, muffle furnace is constructed with the inner walls—between the annular heating portion and the central driving shaft for the stirrer—so thick that air-cooling is sufficient. The heating gases pass first right round the furnace in a flue above the goods, then about three quarters round the furnace in the same direction in a flue under the goods, and leave at a point near where the goods enter, no lower fire passage being provided at that point; the goods travel round counter-current to the gases.

B. M. VENABLES.

[Tunnel] furnaces. H. A. PROCTER (B.P. 291,570, 9.4.27).—Pulverised fuel is used to fire a tunnel kiln and is burned in separate combustion chambers alongside the hottest zone of the tunnel. A method of cooling the walls of the combustion chambers by water is described.

B. M. VENABLES.

Furnaces. C. B. DANN and F. THORESBY (B.P. 290,846 and 291,335, 14.6.27).—(A) A complicated form of hollow fire bridge is described by which steam and air may be introduced between a primary grate space and secondary combustion chamber so as "to assist in the consumption of unburnt gases." In (B) a vaporised light oil may also be supplied through the fire bridge.

B. M. VENABLES.

Regenerative ovens or furnaces. H. SCHMIDT (B.P. 279,505, 24.10.27. Ger., 23.10.26).—A reversing regenerator is filled with loosely packed removable filling which, when choked with dust, is removed from the lower end and replaced by clean at the upper end.

B. M. VENABLES.

Firing arrangement for muffle furnaces. W. J. HARRIS, JUN., Assr. to SURFACE COMBUSTION Co., INC. (U.S.P. 1,672,362, 5.6.28. Appl., 26.9.27).—The muffle is heated by heating gases introduced transversely at the lower part of one side, a portion of the gases passing upwards at the further side and part being diverted back to pass up the side at which they were introduced.

B. M. VENABLES.

[Brick for] furnace structure. H. B. GRONINGER (U.S.P. 1,672,524, 5.6.28. Appl., 20.11.26).—A brick or tile for use in the construction of reverberatory

furnaces comprises a moulded shape of a neutral or basic refractory material enclosed in a metal sheath.

A. R. POWELL.

Heat-exchange cylinder. J. W. OLTMAN, Assr. to BARRETT Co. (U.S.P. 1,672,036, 5.6.28. Appl., 17.1.25).—A stationary cylindrical container is supported by hollow projections which extend axially at both ends. A rotary cylinder surrounds the stationary one, the hollow extensions of which act as bearings. Heat is transmitted from a fluid which is introduced into the stationary container, through one of the hollow supports, to a fluid in the space between the two cylinders.

F. G. CLARKE.

Heat exchanger. W. LONSDALE, Assr. to FOSTER WHEELER CORP. (U.S.P. 1,672,650, 5.6.28. Appl., 27.7.27).—A form of packing joint for baffles in a shell-and-tube heat exchanger comprises a tube split longitudinally down one side and receiving the edge of the baffle.

B. M. VENABLES.

Construction of heat-interchange apparatus. G. CAHILL, Assr. to WINCHESTER REPEATING ARMS Co. (U.S.P. 1,672,708, 5.6.28. Appl., 12.3.26).—A number of thin-walled tubes have enlarged ends fitting together as a honeycomb, and a method of bonding them to each other to form the end wall and to fluid receiving chambers is described.

B. M. VENABLES.

Tubular heat exchangers. CONTRAFLO ENGINEERING Co., LTD., and J. GUNN (B.P. 291,551, 18.3.27).—A screwed joint for securing the tubes in the plates and means for allowing for expansion are described.

B. M. VENABLES.

Water-cooling towers. P. J. ROBINSON and A. T. J. GUERITTE (B.P. 290,321 and 290,553, 10.1.27).—To adjust the water supply uniformly over the area of a water-cooling tower, in (A) the distributing troughs are adjustable as a whole, particularly as to slope, in (B) fixed nozzles in the troughs are provided with easily removable inner nozzles by which the height and aperture of overflow may be varied.

B. M. VENABLES.

High-speed [wet colloid] grinding mills. G. BATER and A. HAMMER (B.P. 290,374, 15.2.27).—The grinding is effected between a fixed stone with a conical space hollowed out on its underface and a lower cone rotated by a vertical shaft; this shaft extends upwards into a feed reservoir and is there provided with stirring arms to prevent settling of the feed mixture. The supply of material is maintained regular by means of air pressure within the closed reservoir.

B. M. VENABLES.

Multichamber mill for grinding materials of all kinds. J. IHLEFELDT (U.S.P. 1,671,291, 29.5.28. Appl.,

4.3.27).—A horizontal, rotary, cylindrical casing is divided into compartments by vertical divisions, each having a segment removed to enable the material being ground to pass from one compartment to the next. The apertures in adjacent divisions are staggered.

F. G. CLARKE.

Rotary mill. J. IHLEFELDT, Assr. to G. POLYSIUS (U.S.P. 1,671,243, 29.5.28. Appl., 31.8.26. Ger., 1.9.25).—The mill comprises a horizontal, cylindrical framework within an axially disposed casing, the space between them forming the sifting compartment, whilst the framework, covered with sifter sections, comprises the grinding compartments. Those sections are secured to ledges on the framework by devices which are operated from the exterior of the casing. The latter has covered openings, through which the sifter sections may be removed.

F. G. CLARKE.

Centrifugal grinding mills. R. M. HUGHES. From BRADLEY PULVERIZER Co. (B.P. 291,696, 28.12.27).—In a mill of the horizontal ring and roller type provided with air separation, streams of air are admitted immediately above the grinding ring and are controlled as to direction by vanes which may be simultaneously adjusted.

B. M. VENABLES.

Roller grinding mills. O. MOOG (B.P. 291,702, 2.1.28).—A servo-feed regulator designed to keep the material in the feed hopper at a constant level.

B. M. VENABLES.

Pulverising mill. W. M. BARKER and L. C. BONNOT (U.S.P. 1,673,483, 12.6.28. Appl., 23.7.24).—Fine powder is exhausted from a ball mill by means of a tube through the axis bent upwards within the mill and having its open mouth protected by a deflector device close to the inner wall of the rotating drum, which deflects the grinding media and ground material downwardly to the rear of the exhaust tube.

B. M. VENABLES.

Pulverising machine. W. L. and H. L. McLAUGHLIN (U.S.P. 1,673,465, 12.6.28. Appl., 5.7.27).—A toothed crusher rotated by a power-driven shaft co-operates with two sets of jaws pivoted about two other shafts and connected together by linkwork.

B. M. VENABLES.

Coal and ore washing plant. C. CLOUWEZ (U.S.P. 1,672,448, 5.6.28. Appl., 4.1.26. Fr., 9.1.25).—The apparatus comprises an inclined fixed table provided with numerous openings, all of which contain a pair of adjustable parallel planes.

A. R. POWELL.

Mixing machines. W. H. SMITH (B.P. 291,509, 1.3.27).—In a mixing machine of the spade type, means for keeping dirt out of the bearings and for tipping are described.

B. M. VENABLES.

Grading and separation of mixed materials. PETRIE & McNAUGHT, LTD., and J. T. CRAVEN (B.P. 290,422, 11.4.27).—A combination of grid tables and conveyor chains for grading mixed materials such as towns' refuse.

B. M. VENABLES.

Continuous centrifugal dryers. T. BROADBENT & SONS, LTD., and W. HALLITT (B.P. 290,432, 30.4.27).—In a separator of the type in which the collected solid matter is discharged by the relative motion of an inner and an outer basket, the outer basket is formed with

non-chokable apertures for the outlet of liquid by constructing it of a number of superposed rings so shaped that the slit-like apertures between the rings enlarge outwardly.

B. M. VENABLES.

Drying apparatus. J. H. RICE (B.P. 290,776, 14.3.27).—An apparatus for the drying of granular material is provided with a fixed bed of wave-like form; in each depression of the waves rotates a wheel carrying a number of buckets which push forward the bulk of the material during the lower part of their rotation and which, at the same time, lift and scatter the material from the highest point they reach. The furnace is heated by a fire the gases from which pass first under the conveyor bed, affording indirect heating and reduction of temperature of the gases, then turn back over the conveyor, giving direct heating of the showered material.

B. M. VENABLES.

Drying ovens. DRYING SYSTEMS, INC., Assees. of G. M. ARGABRITE (B.P. 275,650, 5.8.27. U.S., 5.8.26).—The goods are moved in trays or like containers through one longitudinal tunnel, across to an adjacent tunnel, and back again, by means of synchronised conveyors.

B. M. VENABLES.

Drying drum with distributing apparatus. BÜTTNER-WERKE A.-G. (G.P. 447,529, 1.10.25).—The distributors comprise double hoppers with sloping walls which adjoin at the base, and are so arranged in the drum that the adjacent hoppers of two neighbouring distributors are set opposite to one another.

L. A. COLES.

Drying machine. H. A. HATFIELD (U.S.P. 1,673,241, 12.6.28. Appl., 23.4.27. Can., 26.4.26).—The material passes downwards (in presence of a drying agent) in a zigzag direction through a casing with shelf-like baffles. The baffles are pivoted and balanced, and their slope is controlled by the change in the weight of material discharged from the machine.

B. M. VENABLES.

Apparatus for separating liquids of different sp. gr. W. LINNMANN, JUN. (U.S.P. 1,671,115, 29.5.28. Appl., 22.11.26. Ger., 28.1.26).—The separating tank contains an annular float surrounding a fixed vertical tube, open at the top and bottom, which acts as a guide for the vertical movement of the float. A pipe communicating with the outlet of the tank and extending vertically into the guide tube is closed by a valve-disc supported centrally by the float when the latter is lowered due to a decrease in the buoyancy of the liquid.

F. G. CLARKE.

Apparatus for removing gases from liquids. L. D. MILLS, Assr. to MERRILL Co. (U.S.P. 1,671,601, 29.5.28. Appl., 16.7.25).—The liquid passes from a supply tank into the top of a de-aerating receptacle, in which a vacuum is maintained. The de-aerated liquid is pumped from the bottom of this receptacle through a pipe which passes through the liquid in the supply tank, the pump being also immersed in the latter.

F. G. CLARKE.

Centrifugal filter. W. WEISSGERBER (G.P. 447,583, 16.5.24).—The filtering medium in a centrifugal apparatus in which the material to be filtered, e.g., oil, is driven through a gap between the drum and the cover

is arranged outside the drum and is held in position against the gap by a clamping ring. L. A. COLES.

Rotary vacuum filters and like machines. BRINJES & GOODWIN, LTD., and F. TITTERTON (B.P. 291,148, 24.2.27).—A number of abutting filter chambers are formed round the circumference of an imperforate drum; the filter medium is attached to each chamber individually and may be supported and protected by perforated metal (or textile material) inside and out, the outer protector being preferably a complete cylinder embracing all the sections. B. M. VENABLES.

Hot filtration. C. G. HAWLEY, ASSR. to CENTRIFIX CORP. (U.S.P. 1,668,807, 8.5.28. Appl., 30.7.26).—A vertical cylindrical chamber has means for admitting water and steam at the top in combination with an agitating and whirling device, and for draining the lower part of the chamber, which is separated by a partition from the upper part so as to form a settling chamber in restricted communication with the upper part. A. R. POWELL.

Production of dispersions. L. MELLERSH-JACKSON. From BENNETT, INC. (B.P. 291,502, 28.2.27).—The two liquids, one of which may be a molten thermoplastic substance, are fed in accurately controlled quantities at different places in the upper end of an elongated sloping vessel containing agitators, and the emulsion emerges at the lower end. To prevent the thermoplastic material coagulating into larger drops, counter-current water-cooling may be employed. B. M. VENABLES.

Apparatus for dehydrating liquids. S. M. DICK, ASSR. to INTERNAT. DRY MILK CO. (U.S.P. 1,673,066, 12.6.28. Appl., 22.10.20).—A spray dryer comprising upper and lower drying compartments with a main exhaust space between the two and a subsidiary exhaust also from the lower compartment. Heated air is supplied to both compartments, but the liquid is introduced only in the top one. B. M. VENABLES.

Apparatus for distillation. H. M. GODSEY (U.S.P. 1,673,238, 12.6.28. Appl., 9.7.23).—Oil is supplied to the top of a fractionating column, and falls through it to the still below, where it is distilled by gases of combustion from a furnace which are bubbled through the oil. The distillate passes through a condenser, and the outlet from this divides into a vertical pipe, terminating in a liquid seal for continuous draw-off of liquid, and a pipe leading to a gas exhauster. B. M. VENABLES.

Effecting [continuous] sublimation of volatilisable substances. J. THAME (B.P. 290,849, 23.6.27).—The apparatus comprises a retort with automatic float-operated feed, and thermostatically controlled heating, together with a condenser with internal scraper and air locks or other devices so that the collected sublimate may be removed even though the condenser is under a pressure less than atmospheric. The vapour may be carried forward from the retort to the condenser by a stream of inert gas. B. M. VENABLES.

Crystallisation apparatus. F. JEREMIASSEN (B.P. 290,369, 14.2.27).—Supersaturated liquid is introduced into the crystallising vessel downwards (in a pipe) through a bed of granules, and then turns upward in

contact with the granules. A pipe for the supply of unsaturated or only slightly supersaturated liquid to the space below the granules is also provided.

B. M. VENABLES.

Centrifugal dust separators. ATELIERS J. HANREZ (B.P. 283,146, 29.11.27. Belg., 5.1.27).—The gas current is caused to spin by deflectors, and the centrifugally thrown dust is caught on the outer wall of the apparatus, down which a thin layer of liquid flows. Means for distributing the water and the continuous removal of mud are described. B. M. VENABLES.

Carrying-out exothermic chemical reactions under pressure and at a high temperature. L'AIR LIQUIDE SOC. ANON., ASSECS. OF SOC. CHIM. DE LA GRANDE PAROISSE (AZOTE & PROD. CHIM.) (B.P. 282,658, 13.12.27. Fr., 24.12.26. Addn. to B.P. 268,721; B., 1927, 688).—An excess of gas over that taking part in the reaction is used, the excess being exhausted from the catalysing receptacle at the opposite end to which the gases are admitted, through a tube constructed of heat- and pressure-resisting material. B. M. VENABLES.

Carrying-out exothermic gas reactions. H. HARTER (B.P. 291,253, 4.7.27).—Several methods of effecting uniform distribution of the reacting gases in the mass of a catalyst are described. One form comprises several inlet pipes penetrating to different depths, the outlet from the pipes being through nozzles at the ends, or, at any rate, only in that portion which projects beyond its next shorter neighbour. B. M. VENABLES.

[Automatic] apparatus for the control of temperatures. A. G. S. SANDISON, and ELECTROFLO METERS CO., LTD. (B.P. 290,402, 17.3.27).—A bulb containing a temperature-responsive fluid is connected to a Bourdon tube or other pressure-responsive device which controls a pilot valve through linkwork. The pilot valve may comprise a piston valve which by its longitudinal movement controls a fluid under pressure, and to avoid lag by friction the valve is kept in constant rotation by a jet of fluid acting on a paddle attached to it. The fluid controlled by the pilot valve actuates a piston connected to the fuel-regulating gear, and the piston is also connected to the linkwork, so that when the piston has moved to a new position it returns the pilot valve to normal. B. M. VENABLES.

Thermostats. A. J. THOMAS and A. A. STOW (B.P. 290,423, 11.4.27).—A thermostat, of the type in which an expanding and collapsing capsule obstructs a fuel-supply conduit, is provided with a sliding or rotating carrier, so that any one of several capsules containing liquids which boil at different temperatures may be brought into operation to alter the temperature which is to be maintained constant. B. M. VENABLES.

Thermostats. W. A. VAN BERKEL (B.P. 291,669, 20.10.27).—A rigid reservoir is filled with a temperature-sensitive medium and communicates through a flexible partition to a piston which operates any desired regulating valve. The reservoir is preferably spherical and directly attached to the casing containing the partition and valve. B. M. VENABLES.

Testing the physical properties of materials. R. ESNAULT-PELTERIE (B.P. 289,960, 4.2.27).—The hardness

of two balls of the same material is determined by measuring the electrical resistance of the contact when the balls are pressed together under varying loads. As the load increases, the resistance decreases owing to the greater area of contact, and from the load-resistance curve the hardness may be calculated according to Hertz's equation.

A. R. POWELL.

Hardness testing device. C. H. WILSON (B.P. 290,729, 18.2.27).—A device acting on the same principle as that described in B.P. 277,517 (B., 1927, 863).

B. M. VENABLES.

Colorimeter. R. L. REES (B.P. 291,174, 11.3.27).—Automatic means are described for mixing the liquid to be tested with indicator liquid and transferring the mixture to a colorimetric tube, leaving only the actual comparison of colour to be done by human agency.

B. M. VENABLES.

Apparatus for separating liquids of different sp. gr. W. LINNMAN, JUN. (B.P. 291,608, 9.6.27).—See U.S.P. 1,671,115; preceding.

Introducing and removing goods into or from the working chambers of furnaces. A. SMALLWOOD and J. FALLON (B.P. 291,153, 25.2.27).

Construction of brickwork walls exposed to high temperatures. ÉTABL. F. LABESSE (B.P. 285,050, 10.9.27. Fr., 10.2.27).

Adjustment for retort mouthpiece doors. W. NORRIS (B.P. 291,573, 13.4.27).

Refrigeration processes and apparatus. SILICA GEL CORP., Assecs. of E. B. MILLER (B.P. 266,747, 25.2.27. U.S., 26.2.26).

Evaporators for refrigerating machines. ELECTROLUX, LTD., Assecs. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 275,576, 30.6.27. Ger., 3.8.26).

Measurement of sp. gr. (B.P. 290,913).—See II. Containers (B.P. 283,868).—See VII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Bituminous coal of the Plau series near Dresden. F. FOERSTER and A. LANDGRAF (Brennstoff-Chem., 1928, 9, 169—174).—The bituminous coal of the Döhle basin in the Plau series near Dresden has in all seams the same proportion of coal substance which is in close agreement with that of the upper Zwickau coal. The carbon content of the coal substance is nearly 84%, the maximum given by Bergius for coal formed naturally from cellulose or peat. Under low-temperature coking the coal behaves in the same way as that from Zwickau, Upper Silesia, and the Saar Valley.

A. E. MITCHELL.

Microbiology of coal and its associated strata. I. Microflora of brown coal deposits. R. LIESKE and E. HOFMANN (Brennstoff-Chem., 1928, 9, 174—178).—Microscopical examination of brown coals from several deposits has shown that the microflora is considerably less plentiful than in ordinary soil, there being a general reduction in both the higher and lower plants. Of the higher plants only *Tussilago farfara* was observed. There is a moderate development of Gram-positive soil bacilli

of the phosphorescent group together with several sorts of sporeless fungi. The species observed grow better in ordinary coal than in brown coal.

A. E. MITCHELL.

New theories of coal formation. W. FUCHS (Brennstoff-Chem., 1928, 9, 153—156).—A general discussion of the theories of Bergius (Naturwiss., 1928, 16, 1) and of Taylor (B., 1927, 691; 1928, 288) on the methods of formation of coal.

A. E. MITCHELL.

Use of the Endell heating microscope for the examination of solid fuels. E. BERL and H. SCHILDWÄCHTER (Brennstoff-Chem., 1928, 9, 159—160).—A microscope with an electrically-heated stage has been adopted for the examination of solid fuels. The stage has been fitted with a chamber so that the heating may be carried out in any required atmosphere. With coals it is possible to observe very closely the temperatures at which reactions begin, at which coking is complete, and at which gas ceases to be evolved. A number of coals have been examined in nitrogen, hydrogen, and carbon dioxide, and the effects of these gases on the temperatures at the various stages of the reaction have been determined.

A. E. MITCHELL.

Recognition and determination of the constituents of coal by the "float and sink" method. G. DÖRFLINGER (Arch. Eisenhüttenw., 1927, 1, 3—8, Chem. Zentr., 1927, II, 1527).—The individual constituents of a coal may be separated by the use of a series of solutions of graduated density. From the results obtained, a "value number" of the coal may be obtained which is characteristic for every type of coal, and from which conclusions may be drawn as to the value of the coal for the manufacture of gas and coke.

A. R. POWELL.

Analytical characterisation of coals. W. FUCHS (Brennstoff-Chem., 1928, 9, 198—200; cf. B., 1928, 145).—In addition to carboxyl groups (present only in geologically recent coals), which can displace acetic acid from calcium acetate, old and young coals contain acidic hydroxyl groups which can be titrated by means of aqueous or alcoholic alkali hydroxide. Determination of the "hydroxyl number" serves to characterise coals analytically. The sample, corresponding with about 0.3 g. of dry coal, is shaken and kept with 50 c.c. of 0.1N-sodium hydroxide solution; a few c.c. of 10% barium chloride solution are added and water to a total of 100 c.c., and a filtered aliquot portion is titrated using 0.1N-sulphuric acid and phenolphthalein. A blank experiment is also carried out. Alternatively, a sample corresponding with about 1 g. of dry coal is boiled for 30 min. with 50 c.c. of 0.1N-alcoholic potassium hydroxide, and the solution titrated as before, after adding sufficient alcohol to keep the alcohol concentration not less than 75% at the end of the titration. Since the same results are obtained working in cold or in boiling alcoholic solution, humic anhydride, if present, does not take part in the reaction. Eight coals have been investigated. The hydroxyl numbers determined in aqueous solution were mostly smaller than those found in alcoholic solution, and showed greater differences between different coals. The hydroxyl number is greater in recent coals than in older ones.

W. S. NORRIS.

Fusion of coal, coke, and motor fuel by sodium peroxide. G. E. MABEE (Ind. Eng. Chem., 1928, 20, 644—645; cf. Selvig and Fieldner, B., 1927, 513).—In the determination of sulphur in coke by means of sodium peroxide according to usual methods unburned material occurs to the extent of about 2%; addition of benzoic acid etc. to the charge helps to supply the volatile matter which anthracite and coke lack. Alternatively, it is recommended that the cartridge containing the charge be placed in a muffle at 800° for 2 min., thus ensuring even heating and better combustion. Sulphur in volatile substances, e.g., petrol, benzene, etc., is determined by igniting a mixture of the volatile material and sodium peroxide in a nickel crucible which stands in water to half its height. A copper canopy covers the crucible, and spontaneous ignition is started by introducing 0.5 c.c. of methyl alcohol into the charge. For a satisfactory energetic combustion, about 15—18 g. of sodium peroxide and 1 c.c. of the liquid fuel to be tested should be used. C. A. KING.

Humic acids. Pyrohymatomelanic acid, a new compound isolated from Merck's humic acid. D. J. W. KREULEN (Brennstoff-Chem., 1928, 9, 197—198).—Merck's acidum humicum contains an insignificant quantity of water-soluble humic acid (fulvic acid), and about 27% of alcohol- and phenol-soluble, yellowish-brown hymatomelanic acid, 9% of brownish-black pyrohymatomelanic acid (insoluble in 95% alcohol or ether, soluble in phenol), and 64% of darker-coloured, insoluble, residual humic acids. The new acid is intermediate between hymatomelanic acid and the insoluble humic acids in ease of precipitation from an alkaline solution by means of mineral acids. A similar brown granular compound is precipitated when a solution of hymatomelanic acid in 95% alcohol is boiled; if the operation is too prolonged the precipitate is also insoluble in phenol (cf. Erdmann and Dolch, "Chemie der Braunkohle," 1927, 124). W. S. NORRIS.

So-called "nitrohumic" acid. W. FUCHS (Brennstoff-Chem., 1928, 9, 178—182).—The product obtained by treating humic acid, from brown coal, with nitric acid and its methyl derivative have been examined and their properties compared with "nitrohumic" acid, prepared from carbamide, and its nitroso-derivative. "Nitrohumic" acid is shown to have properties similar in all respects to the nitroso-derivative from carbamide. Mol. wt. determinations combined with corresponding determinations of the equivalent weight lead to the suggestion that the compound is an isonitrosoketotrihydroxytetracarboxylic acid with a mol. wt. of 1300 (approx.). A. E. MITCHELL.

Carbonisation. III. Temperature, size of coal, blending with coke and inorganic compounds. 20TH REP. OF RES. SUB-COMM. OF GAS INVESTIGATION COMM., INST. GAS ENG. (Gas J., 1928, 182, 883—888; cf. B., 1927, 592).—Experiments on carbonisation of a moderate-coking Nottinghamshire coal have been continued, the apparatus used being the same as that previously described except that improvements in the scrubbing and purifying systems have been made. With temperatures below 820° the yields of coke, tar, liquor, and gas were only slightly affected by grading, but at

higher temperatures the calorific value of the gas was lower and the tar yield greater when large coal was used. Although the total volume of gas produced did not vary greatly, the initial rate of evolution was lower with small coal and the total duration of carbonisation was considerably prolonged. The retort temperature influences the period of carbonisation, so that 2.5 hrs. at 915° produce the same effect as 10 hrs. at 630° and 20 hrs. at 525°, and the yield of therms per ton of coal falls from 75.8 at 979° to 4.8 at 344°. The composition of the gas varies with rising temperature, carbon dioxide decreasing, hydrogen and carbon monoxide increasing, and unsaturated hydrocarbons reaching a maximum at about 450°. Blending with coke produced no beneficial results, but the mixture was not briquetted, and the coal was one of only moderate coking properties. Addition of 2% of sodium or calcium carbonate or iron oxide all gave increased thermal yields of gas at temperatures above 815°, and formation of ammonia varied in the same direction. Preliminary tests on the reactivities and physical properties of the cokes show that lowering the temperature of formation increases the reactivity to steam and to carbon dioxide, and decreases the mechanical strength, but does not affect the ignition temperature except in cases where inorganic oxides have been added to the coal.

R. H. GRIFFITH.

Factors influencing reactivity of coke. I (a). Carbonising temperature and heat-treatment in nitrogen. J. A. SUTCLIFFE and J. W. COBB. I (b). **Heat-treatment in hydrocarbon and other gases.** F. J. DENT and J. W. COBB (Gas J., 1928, 182, 946—948, 948—951).—I (a). The addition of inorganic oxides to a coal has been shown to influence the properties of the coke formed by its carbonisation (cf. preceding abstract, also B., 1927, 833), and the effects obtained by mixing sodium, calcium, and potassium carbonates and iron oxide with Sharlston Wallsend coal have been studied. At 500° the untreated coal gave a spongy coke, and that produced by addition of calcium carbonate was similar, whilst those containing other oxides were finely porous and homogeneous. At 1100° all the cokes except that containing potassium were hard. Study of the reaction of these cokes with steam showed that those obtained at 1100° were the least reactive, whilst 500° cokes did not react as extensively as those produced at 800°; in all cases the influence of inorganic oxides was marked, particularly with sodium. Further experiments with a coke formed at 500° showed that heating in nitrogen for several hours at 1000° or gasifying in steam at 1000° produced no material change in its reactivity, but prolonged heating (50 hrs. at 900°) of 800° coke reduced its activity considerably.

I (b). Coke obtained from Sharlston Wallsend coal, containing 1.3% of ash, at 800° for 6 hrs., was heated for 1 hr. at different temperatures in hydrogen, carbon monoxide, methane, and ethylene, and changes in the reactivity of the coke with carbon dioxide were investigated. Heating in hydrogen or carbon monoxide at 900° led to slight loss of activity, whilst the effects with methane were greater; the influence of ethylene was much more pronounced and appeared at lower temperatures. The reduction of reactivity was also more

persistent in the last case, and decomposition of the ethylene was detected; at 900° tarry matter was formed which contained naphthalene, and a hard carbon deposit was visible. The rate at which the coke regains its activity on prolonged heating in carbon dioxide appeared to depend on the extent to which hydrocarbon decomposition had occurred.

R. H. GRIFFITH.

Influence of inorganic constituents in the carbonisation and gasification of coal. II. Liberation of sulphur. J. J. PRIESTLEY and J. W. COBB (Gas J., 1928, 182, 951—954. Cf. B., 1925, 620; 1926, 1002; 1927, 833).—Experiments have been carried out with coals to which inorganic substances were added, in nitrogen and hydrogen; charges of about 20 g. of Sharlston Wallsend coal, heated in a Morgan tube, were used. The "pure" coal in nitrogen gave off 42% of its sulphur by heating to 1000°, of which the bulk appeared at about 500°, whilst in hydrogen the evolution of hydrogen sulphide continued at higher temperatures and reached 68% at 1000°. With 5% of iron oxide added to the coal only 9% of the sulphur was liberated in nitrogen, but in hydrogen rapid decomposition occurred from 800° upwards, amounting to 64% at 1000°. When lime was added to the coal the decomposition in nitrogen or hydrogen was less than 3%. Mixing with sodium carbonate gave increased liberation of sulphur in nitrogen until the process was interrupted by the formation of sodium oxide at about 600°, whilst in hydrogen similar results were observed to a greater degree.

R. H. GRIFFITH.

Effect of alkali-metal compounds on combustion. C. A. THOMAS and C. A. HOCHWALT (Ind. Eng. Chem., 1928, 20, 575—577).—Since a rather concentrated solution of sodium potassium carbonate in water has been found to extinguish flames, the effect on combustion of a large number of compounds in solution has been studied. A jet of the solution under trial was directed on to the internal wall of a fire pan containing ignited gasoline, the whole being contained in a draught-proof chamber. The temperature of the fire pan was allowed to reach 270° before the extinguishing jet was opened, and all conditions were adjusted accurately for comparative purposes. With few exceptions every element capable of forming a water-soluble salt was studied. Practically all salts of the alkali metals exhibited this fire-extinguishing effect, which is described as "catalytic," although quantitative results were influenced very decidedly by the radicals attached to the metal, the effect also increasing with increase in the at. wt. of the metal. The extinguishing effect of compounds of alkali metals, e.g., halides, was increased considerably by the presence of combined oxygen, though the presence of an alkali metal appeared to be essential. Organic compounds of the alkali metals were effective extinguishers, the degree being generally proportional to their oxygen content. Although sodium potassium carbonate was remarkably effective, carbonates of other metals gave a negative result, and even ammonium carbonate, which dissociates easily, failed entirely to extinguish the fire. In working with the minimum effective concentrations of the alkali metals, these weak solutions were instantly effective when the tem-

perature of the sides of the fire pan above the gasoline level was 200—325°; above that range an interval occurred before the fire was extinguished, and apparently the solution cooled the pan to the effective range before the "catalytic" effect was possible.

C. A. KING.

Effect of physical characteristics of coke on reactivity. J. D. DAVIS and D. A. REYNOLDS (Ind. Eng. Chem., 1928, 20, 617—621).—Low-temperature cokes are of a distinctly higher order of reactivity than those produced at higher temperatures, though little difference was noted between similar cokes from different coals, and variation of the method of high-temperature production within the range of American practice has no pronounced effect on reactivity of the coke. No close relation exists between the properties of lower bulk density and higher reactivity of low-temperature cokes, and the increase in reactivity corresponding with a high content of volatile matter is probably incidental. Reactivity varies inversely with the size of test particles and generally directly as the adsorptive power. With high-temperature cokes external surface has a greater effect on reactivity than capillary surface. Different coke particles of the same sample show different reactivities, the duller portions from the centre of the piece being more reactive than the brighter portions.

C. A. KING.

Properties of coke. H. GREGER (Brennstoff-Chem., 1928, 9, 156—159).—An apparatus for studying the ignition and burning of solid fuels is described. Tests on different varieties of coke show that the inception and maintenance of the combustion depend not only on the ignition temperature but also on the oxygen supply and its concentration, the heat loss from the combustion chamber, and the nature of the igniting influence. It is proposed that the term "combustion threshold" embodying all these factors should be applied as indicating the suitability of a fuel for burning in any particular manner.

A. E. MITCHELL.

Firedamp explosions: projection of flame. II. M. J. BURGESS (Safety in Mines Res. Bd., Paper No. 42, 1928, 8 pp.; cf. B., 1926, 906).—The distance of projection of flame from the explosion of mixtures of methane and air has been determined in a gallery 7½ ft. in diam., with columns of explosive mixture of 26¾ and 53¾ ft. in length, respectively, and with varying proportions of methane in the mixtures. With the shorter column the distance of projection was 60 ft. with a mixture containing only 6.3% of methane. The maximum projections, obtained with mixtures containing about 10.5% of methane, were 120 and 220 ft. with the shorter and longer columns of explosive mixture, respectively. These distances are somewhat shorter than those obtained in laboratory experiments or calculated theoretically.

A. B. MANNING.

Disposal of liquor effluents from gas works. 2ND REP. INST. GAS ENG. LIQUOR EFFLUENTS RES. COMM. (Gas J., 1928, 182, 1016—1018; cf. B., 1927, 864).—The condensation of tar and liquor from a vertical retort setting at Hinckley has been studied. The gas was passed through air-cooled and water-cooled condensers, an exhauster, Livesey washers, and tower

scrubbers before entering the oxide boxes; the tar and liquor from all points ran into a common well from which liquor was removed periodically or pumped to scrubbers. 17.2 gals. of tar and 32 gals. of liquor were obtained per ton of coal, and the oxygen-absorption value of the liquor was 950 pts. per 100,000, of which the majority was due to phenols and tar acids, but a great increase in the thiocyanate and thiosulphate content occurred in liquor which had been used in washers after storage in contact with air. The content of higher tar acids was reduced by introduction of a tar extractor in the foul main, and still better results would be anticipated if more tar could be condensed before separation of any liquor. Thiocyanate and thiosulphate increased sevenfold when air was admitted at the inlet to the condensers, so that the presence of these substances can be lessened by adding air only at the oxide boxes, and also by reducing the temperature of the circulating liquor. The final effluent from the ammonium sulphate plant was 1.4 times the volume of liquor handled, and was about 1% of the total dry-weather sewage; this figure might be reduced to 0.5% by provision of suitable balancing tanks. Disposal of effluent, under careful control, into sewage is considered satisfactory, whilst separate disposal of devil-liquor, by means of chimney-spraying etc., reduces the work of effluent purification by about 20%.

R. H. GRIFFITH.

Products of combustion from typical gas appliances. III. Appliances using free-burning flames. 19TH REP. OF RES. SUB-COMM. OF GAS INVESTIGATION COMM., INST. GAS. ENG. (Gas J., 1928, 182, 880—882; cf. B., 1927, 593).—As in the previous work, iodine pentoxide has again been used to determine the carbon monoxide produced, but the gas was first freed from carbon dioxide, and from oxides of nitrogen by condensation in liquid air. In all cases of isolated burners such as Bunsen, Argand, or Méker, the production of carbon monoxide was very small, being less than 3 pts. per 10,000 of gas used. A condensing-type flueless heater gave 0.1 pt. at its rated capacity and only slightly more with excessive gas supply; a 3-gal. water heater with automatic control gave 2.0 pts. at low gas rates, but with high water and gas rates only 0.1 pt. A geyser burning 136 cub. ft./hr. was fitted with flue pipes below the baffle, of varying lengths, and the carbon monoxide fell rapidly from 120 pts. at about 10 in. to negligible amounts at 5 ft. A Junkers calorimeter, in which the Bunsen flame burns in a water-cooled chamber, gave lower results than a similar flame in an open space. Extensive experiments have been made with a gas oven heated by two side burners; the carbon monoxide content is found to fall, whereas the flue temperature and amount of carbon dioxide rise as the gas consumption increases. Preliminary tests on gas fires with the new technique show that, when properly adjusted and running at full capacity, not more than 50 pts. of carbon monoxide should be formed per 10,000 pts. of gas burned.

R. H. GRIFFITH.

Design of gas-burning equipment. N. T. BRANCHE (Chem. Met. Eng., 1928, 35, 357—360).—A short review of fundamental principles.

Comparison of mechanical tar washing and electrical precipitation processes. F. SEIDENSCHNUR and E. GROH (Brennstoff-Chem., 1928, 9, 188—193).—The performances of a Theisen mechanical tar washer and a single-stage electro-filter of the Siemens-Schuckertwerke type have been compared when working on gas obtained from central German brown coal briquettes in a revolving-grate gas generator with a low-temperature carbonisation attachment. The electro-filter removed 99.4% of the tar whilst the Theisen washer, using as wash water the brown coal carbonisation water, removed only 95.3%. The tar obtained by the two methods of recovery was of good quality and of almost identical properties. The experiments indicated that by the correct choice of working conditions and type of generator complete recovery of tar and light oils should be possible.

A. E. MITCHELL.

Determination of water [in coal, tars, and oil] by distillation with xylene or tetrachloroethane. K. SCHAEFER (Chem.-Ztg., 1928, 52, 408).—The author discusses various modifications of his original apparatus (B., 1924, 973) which have been suggested by others, and describes an improved form which can readily be adapted for use with tetrachloroethane instead of xylene.

W. J. BOYD.

Conversion of coal into oil by the Bergius method. J. I. GRAHAM and D. G. SKINNER (J. Inst. Petroleum Tech., 1928, 14, 78—93).—Coal samples (60-mesh I.M.M.) were treated in a 3-litre steel autoclave, gas-heated and rotated at about 60 r.p.m. In all experiments 200 g. of coal were used suspended in 100 g. of phenol. The maximum pressure during hydrogenation was 145—150 atm., the temperature was kept at about 430°, and the total duration of heating was 8 hrs. carried over two days, the hydrogen being renewed. The products of reaction were first distilled in an aluminium retort up to 150°. The light spirit fraction was washed with dilute caustic solution and the remaining products were further treated with chloroform, thus yielding (a) phenol-insoluble, (b) phenol-soluble, chloroform-insoluble, and (c) phenol-soluble, chloroform-soluble fractions. Products (c) were further separated into fractions distilling up to 300°, and between 300° and 450°. The results show yields varying from 5.8 to 109 gals. of total oil per ton of coal, corresponding to petrol yields of 2.8 to 23.9 gals. per ton of original coal. The addition of ferric oxide as catalyst to the original charge increased the petrol yield from an equivalent of 9.5 to 15 gals. per ton of coal; there was also an increase in tar oil and amount of aqueous liquor produced, but the yield of product (b) was practically unaffected. In general, the yields of tar oil (product c) are less than those obtained by Bergius. The effect of raising the hydrogen pressure was to increase the yield of tar oil. The tables indicate that the solid residue left after hydrogenation averages about 20%, and the yield of gaseous products is about 20% by wt. of the original coal. The general conclusions are that petrol suitable for internal-combustion engines may be obtained, on an average, in amount up to 25 gals. per ton of coal treated. The value of the heavier oil, however, is not so assured. Certain coals have proved to be more suitable for treatment than others, and these appear to

fall into the parabituminous division of the Seyler classification. Those having a carbon:hydrogen ratio between 15.5 and 16.5 appear to give the highest oil yields. From calculations of the costs of the process it is considered that such coals could possibly be worked at the present time with profit. H. S. GARLICK.

Vapour-phase oxidation of aromatic hydrocarbons and of petroleum distillates. A. R. BOWEN and A. W. NASH (J. Inst. Petroleum Tech., 1928, 14, 106—114).—The possibility of the conversion of certain petroleum distillates by vapour-phase oxidation was considered by first investigating its effect on pure aromatic hydrocarbons. In the apparatus used, air was measured in a wet meter and drawn through scrubbing towers containing caustic potash and strong sulphuric acid, respectively, then passed through a copper worm, and bubbled through the liquid under test, both being immersed in a thermostat. The vapour-enriched air was led into the reaction tube resting in a stout iron tube in an electric furnace. The hard glass reaction tube contained 60—70 c.c. of 5—10-mesh aluminium grains to serve as preheater, followed by the catalyst, consisting in each case of 60 c.c. of similar aluminium grains, but coated with vanadium pentoxide. The exit gases were led through a water-cooled condenser to an ice-cooled flask, then through two water washers, and finally through two U-tubes filled with activated charcoal. 50 c.c. of pure toluene were run into the "carburettor" kept at 50°, and the enriched air passed over the catalyst maintained at 400° at the rate of 12 litres/hr. In all 127.5 litres of air were used. At the cool end of the exit tube a mixture of crystals separated which were identified as anthraquinone (0.3 g.), benzoic acid (4.87 g.), and 0.37 g. of the mixed crystals. An oil condensate (24.7 c.c.) and reaction water (7.3 c.c.) were obtained. From 18 c.c. of the oil condensate, on fractionation, were obtained 16 c.c. volatile up to 115° and a residue of 2 c.c. of crude benzaldehyde. The aqueous distillate had an acid reaction, decolorised acidic potassium permanganate solution and bromine water, and reduced ammoniacal silver nitrate. The activated charcoal tubes gave 6.5 c.c. of unchanged toluene. A further experiment with "toluol benzine" ex Borneo crude gave substantially similar results. *n*-Hexane was found to give oxidation products under similar conditions to the above. Out of 50 c.c. of this hydrocarbon 45.9 c.c. were recovered as an oil. H. S. GARLICK.

Sulphur compounds in transformer oil. E. FERBER (Z. angew. Chem., 1928, 41, 680—682).—The complete removal of sulphur from the heavier distillates of petroleum or lignite tar, which is necessary before hydrogenation, is effected by treatment with metallic sodium. The carbonaceous residue was acidified with dilute hydrochloric acid and extracted with light petroleum, from which a yellow oil was obtained by evaporation. This proved to be a mixture of disulphides, from which, by reduction, amyl, heptyl, and octyl mercaptans were isolated. C. IRWIN.

Designing equipment for chemical treatment of oil distillates. J. C. MORRELL and D. J. BERGMAN (Chem. Met. Eng., 1928, 35, 291—295).—To proportion

the reagents when blow-cases are used under various differential pressures, several sizes of orifices must be used. A table and chart are given showing the rates of flow and pressures with different diameter orifices. A differential pressure across the orifice of 6—20 lb. is recommended. Pumps for acid, caustic, and plumbite feed should be of acid-resisting bronze or of cast iron and steel with liners, and should be of the positive piston, duplex or rotary type having an approximate capacity of 10 brls. per hr. Recirculating pumps of the rotary gear or centrifugal types should pass 20 brls. per hr. Both types should have by-pass arrangements. The inlet to the settling tank should be made at the separating level, and tangential to the tank, in order to set up a whirling motion which assists in settling. The water spray may be a cross-pipe with perforations on one side of each pipe. An orifice mixer for water washing is not recommended. A system for removing hydrogen sulphide from cracked distillates is described consisting of a water wash followed by a caustic wash. The same equipment can serve for pretreatment of cracked distillates with plumbite solution. The design of a 1000-brl. agitator for batch-treating systems is discussed in which a centrifugal pump is installed in connexion with recirculation lines for drawing off separately the reagents and pressure distillate, the relative proportions being controlled by a valve. The reagent and pressure distillate enter the suction side of the line leading into the top of the agitator, the discharge being below the level of the oil.

H. S. GARLICK.

Operating a continuous plant for refining [petroleum] distillates. J. C. MORRELL and D. J. BERGMAN (Chem. Met. Eng., 1928, 35, 350—354).—A continuous treating plant consists of mixers for acid, sodium plumbite, and caustic soda, with intermediate settling tanks and provision for water-washing. For mixing, orifice columns followed by baffled columns are used. The latter prolong the time of contact and allow of recirculation of the reagent if desired. The proportioning of the reagents is effected by pumps. Plant dimensions are discussed in detail. Lead-lined pipe or chromium alloys are available for the acid used. Stoneware towers and stoneware-lined pipes are suggested for such reagents as cupric chloride. The use of separating sumps in which oil may be collected from aqueous drainage is desirable. C. IRWIN.

Simplified calculations in design of natural gasoline absorbers. W. K. LEWIS (Chem. Met. Eng., 1928, 35, 93—95).—A method of computation of continuous counter-current systems for the absorption of any single component in a gaseous mixture by a non-volatile liquid absorbent in which the other components of the gas are substantially insoluble is applied to the absorption of the large number of components present in natural petroleum. In a system in which a single component is being absorbed, an equation in which the gaseous phase is linear in terms of the corresponding concentration in the liquid phase may be obtained representing the operating conditions at every point in the absorber. The slope of this line (the "operating" line) is L/G , the ratio of absorbing liquid to inert gas

submitted to treatment. From this ratio the operating conditions throughout the absorber can be determined. The distance between the operating line and the equilibrium curve represents the driving force producing absorption, and is therefore a quantitative measure of the rate of absorption in the unit. The operating lines for each component of natural gas, considered separately, are parallel straight lines. The equilibrium curves are straight lines through the origin with the slope T/π , where T is the temperature of absorption and π the operating pressure on the unit. For high-boiling constituents the driving force becomes very large at the rich end of the diagram but very small at the lean end, accounting for the difficulty in completely removing the high-boiling very soluble constituents from the gas by liquid absorption, despite their solubility.

H. S. GARLICK.

Continuous treatment of light oil products.

A. A. KECHER (Azerbejdianskoye Neft. Khoz., 1927, No. 10, 77—82).—Polemical. CHEMICAL ABSTRACTS.

Distillation of oil products in a stream of different gases. N. TSCHERNOSHUKOV (Neft. Khoz., 1927, 13, 63—65).—The speed of vaporisation of Baku or Grozny petroleum (b.p. 100—150°) is unchanged when the distillation is carried out in hydrogen, carbon dioxide, nitrogen, or air. CHEMICAL ABSTRACTS.

Determination of water and mud in oil products.

T. A. SELSKI (Neft. Khoz., 1927, 13, 623—631).—The water layer is drawn off and the water-petroleum emulsion is broken up by shaking. The use of a centrifuge is desirable for the separation and for the determination of mud. CHEMICAL ABSTRACTS.

Determination of sulphur in crude petroleum and its products. M. KORSKOV (Neft. Khoz., 1928, 14, 68—81).—The funnel of the absorption apparatus is provided with an inverted rim to collect water condensing on the walls; this water contains 15% of the sulphur dioxide produced. The U-tube of the absorption column is fitted with a draining cock, and the lamp has a ground-in cover and stopper to prevent evaporation. The titration is carried out electrometrically, using platinised platinum electrodes, at $5 - 6 \pm 0.2^\circ$ above the ordinary temperature.

CHEMICAL ABSTRACTS.

Determination of sulphur in volatile fuels.

H. T. KENNEDY (Ind. Eng. Chem., 1928, 20, 201—202).—A modified type of test lamp is described (cf. Lomax and Bevan, B., 1925, 124), in which the fuel is vaporised in a carburetting device and burned, and the oxides of sulphur so produced are absorbed by a sodium carbonate solution of known concentration. Advantages of speed and accuracy over other methods are claimed.

H. S. GARLICK.

Separation of lead tetraethyl from solution in petroleum spirit. F. W. TOMS and C. P. MONEY (Analyst, 1928, 53, 328—329).—When "ethyl petrol" is saturated with sulphur dioxide it deposits after keeping a white solid, which on treatment with concentrated sulphuric and nitric acids yields a white precipitate. This was weighed and further purified by dissolution in hot ammonium acetate. Assuming lead tetra-

ethyl to have $d 1.62$, then 0.0833 vol.-% was found in a sample. Petrol extraction followed by treatment with sulphur dioxide is suggested in the case of other suspected materials.

D. G. HEWER.

Distribution of sulphur in [American] oil shale.

II. E. P. HARDING and W. H. DUMKE (Ind. Eng. Chem., 1928, 20, 164).

Premature failure of refractories. DALE.—See VIII. **Welding with coal gas.** MÜLLER and BIBUS.—See X. **Carbon black for rubber.** GOODWIN and PARK.—See XIV.

PATENTS.

Coke-oven heating. Retort oven. H. KOPPERS, ASSR. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,671,194—5, 29.5.28. Appl., [A] 5.7.21, [B] 31.8.21. Ger., [A] 4.4.17, [B] 4.3.19).—(A) The heat economy of a regenerative, gas-fired, coking furnace is improved by shutting off the supply of gas at a suitable moment during carbonisation and circulating air through the combustion chamber in such a way that the air, acting as a heat distributor, transfers heat from the hotter to the relatively cooler parts of the combustion chamber and regenerators. The supply of gas to the combustion chamber is then resumed. (B) An oven plant comprises vertical retort chambers, parallel vertical heating flues, regenerators so arranged that the hot waste gases flow through them from top to bottom and the combustion gases in the reverse direction, an upper horizontal channel connecting the heating-flues with the top of one of the regenerator groups, and a lower horizontal channel and guide channel connecting the heating-flues with the other regenerator group.

A. B. MANNING.

Multiple-chamber oven chiefly for distilling at low-temperature carboniferous material. SOC. DE RECHERCHES ET DE PERFECTIONNEMENTS INDUSTRIELS (B.P. 279,130, 17.10.27. Ger., 15.10.26).—The material to be carbonised is heated in a series of chambers in succession, by the passage through them of a gaseous heating medium, e.g., steam. Means are provided for controlling the temperatures employed, which vary from one chamber to the next in any desired manner. The material remains stationary during each stage of the heating process. The chambers are arranged on the same level and the floors are vertically movable, e.g., by jack pistons, so that the charge in each chamber, which is contained in a tub, may be lowered, moved horizontally, and raised into the next chamber.

A. B. MANNING.

Ovens for treating fuels or fuel-containing materials by heat. O. HELLMANN (B.P. 290,838, 2.6.27. Addn. to B.P. 272,493; B., 1928, 240).—Hollow spaces with perforated walls are provided in the compartments containing the material, in order to facilitate the escape of the volatile products evolved. Also, to enable uniform heating in the acute-angled heating chambers of the ovens with annular platforms to be secured, the heating agent is caused to enter the flues not only at the outer periphery, but also at points at distances away from the outer periphery.

A. B. MANNING.

Production of solid, smokeless fuel from carbonaceous material. A. E. WHITE. From ILLINOIS

ANTHRACITE CORP. (B.P. 291,206, 26.4.27).—The material is fed at a comparatively fast rate into a producer, in which a relatively shallow fuel bed of constant depth is maintained. The material, which is continuously agitated, is subjected to restricted combustion so that only part of the volatile matter and none of the fixed carbon is consumed. The product is continuously discharged. A producer, suitable for carrying out the process, is of the rotary-shell type, with an idly rotating hearth for supporting the bed of material, a stirrer, and a stationary discharging mechanism. A. B. MANNING.

Fuel material [briquettes]. T. NAGEL (U.S.P. 1,666,663, 17.4.28. Appl., 31.10.24).—A binding material for anthracite briquettes is obtained by dextrinising a mixture of equal parts of flour waste, 20% phosphoric acid, and water. Powdered anthracite is mixed with 10% of the resulting solution, briquetted, and baked at 300°.

A. R. POWELL.

Apparatus for measuring sp. gr., adapted for determining the ash or mineral content of coal or other samples. K. KEGEL (B.P. 290,913, 19.11.27).—A device for measuring the volume of the coal sample by displacement of water is incorporated with an automatic weighing machine, which is provided also with a computing scale to indicate not only the weight of the sample, but also the ash content according to its sp. gr.

A. B. MANNING.

Production of a gaseous mixture from pulverised coal. F. HERZBERG (B.P. 266,354, 16.2.27. Ger., 19.2.26).—The powdered coal is charged into an externally heated vertical retort down a tube which terminates a considerable distance below the gas outlet, so that no coal is carried away by the gas. Superheated steam is introduced at the bottom of the retort, and the process is conducted so that as far as possible all the fuel is gasified. The retort may be enlarged in cross-section at the level of the coal supply pipe in order to reduce the relative velocity of the gas and coal at that point. A gas of high calorific value is produced.

A. B. MANNING.

Process for gas making. H. R. BERRY (U.S.P. 1,672,052, 5.6.28. Appl., 5.4.28).—The heat produced in air-blasting one fuel bed is used to distil the volatile matter from a second fuel bed. The air blast is then discontinued, and a combustible gas is produced by the passage of steam, together with the volatile products distilled from the second fuel bed, through the first fuel bed.

A. B. MANNING.

Separation and recovery of olefines from gases containing same. SYNTHETIC AMMONIA & NITRATES, LTD., and G. F. HORSLEY (B.P. 291,186, 30.3.27).—Ethylene or other olefines are separated from coal distillation gases etc. by scrubbing the gas, from which the sulphur compounds and ammonia have been removed, with an acid solution of silver nitrate, either under ordinary or increased pressure; the ethylene is subsequently recovered by heating the solution. A solution 0.3N with respect to nitric acid and containing 200 g. of silver nitrate per litre is suitable for the purpose. The temperature of the solution is preferably maintained at 0–10° during absorption and at 50–70° during regeneration.

A. B. MANNING.

Production of carburetted water-gas. C. W. ANDREWS (U.S.P. 1,672,109, 5.6.28. Appl., 3.4.22).—Carbonised material is air blasted and the blast gases are passed through a heat-absorbing structure surrounding a coking retort. The material is then steamed and the hot water-gas, after being further heated by passage through the structure previously heated by the blast gases, is passed through coal in a separate coking retort. The coal is thereby distilled and the mixture of water-gas and coal gas produced is carburetted by the addition of enriching material and passage through a suitable cracking and fixing chamber.

A. B. MANNING.

Manufacture of producer gas and water-gas. HUMPHREYS & GLASGOW, LTD., Assees. of SOC. DE CONSTRUCTION D'APPAREILS POUR GAZ A L'EAU ET GAZ INDUSTRIELS (B.P. 285,004, 6.12.27. Fr., 8.2.27).—The fuel bed in a suitably designed producer can be subjected to down-steaming as well as up-steaming; oxygen is admitted with the steam either during the up- or the down-run or both. Thus, in one method of working, excess oxygen is injected during the up-run, whilst steam only is injected during the down-run.

A. B. MANNING.

Apparatus for the splitting of acetylene. P. BURGER, Assee. of "HYDROCARBON A.-G. F. CHEM. PROD." (B.P. 264,500, 12.1.27. Ger., 12.1.26).—The explosive decomposition of acetylene is carried out in a long horizontal cylindrical pipe. To minimise damage in case of accidents the heavy main fittings are fixed in the longitudinal direction of the pipe, and protecting walls of concrete, iron, sand, etc. are erected a short distance from them.

A. B. MANNING.

Composition for use in substitution for asphalt and bituminous materials and for electrical insulation. G. T. FUERY. From AUSTRALIAN BITUMENOUS COMPOUNDS, LTD. (B.P. 290,886, 20.9.27).—The base of the composition consists of a mixture of pulverised oil shale and tar. With this is incorporated a suitable filling material for the production of road coverings, damp courses for buildings, electrical insulating materials, etc.

A. B. MANNING.

Protecting the interior of an oil-cracking retort. A. C. HOLZAPFEL (B.P. 291,585, 30.4.27).—The interior of the retort is coated with a cementitious composition which is allowed to set. A suitable composition consists of 45 pts. by wt. of water-glass, 28 pts. of water, 5 pts. of magnesite, 2 pts. of slaked lime, and 10 pts. of fire-clay, with or without the addition of a pigment such as titanium dioxide.

C. O. HARVEY.

Pressure-still process. C. B. BUERGER, Assr. to GULF REFINING Co. (U.S.P. 1,672,801, 5.6.28. Appl., 26.4.27).—In a pressure still, clogging of the outlet for tar and residuum is prevented by intermittently forcing a solvent through this outlet in a direction opposed to that of normal flow.

C. O. HARVEY.

Manufacture of liquid hydrocarbons of low b.p. from olefines. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 291,137, 22.2.27).—Hydrocarbons of the naphthene and aromatic series are obtained from olefines or gases containing them by the application of heat and pressure in the presence of suitable cata-

lysts. At 300–400° the products consist principally of liquid olefines, whilst at higher temperatures (up to 900°) aromatics and naphthenes are the principal products. Suitable catalysts are metallic gold, silver, or copper, in such form as to exhibit coherent surfaces, *e.g.*, a copper tube, oxygen-containing compounds of the alkali metals, oxyacids of phosphorus, boron, and antimony, or their salts, excepting, in the case of gases containing carbon monoxide and hydrogen, those catalysts capable of forming methyl alcohol. The formation of methane may be checked by adding a gas containing methane. C. O. HARVEY.

Production of low b.p. and other hydrocarbons by the destructive hydrogenation of coals, tars, mineral oils, etc. I. G. FARBENIND. A.-G. (B.P. 273,337, 27.6.27. Ger., 26.6.26).—The starting materials, if liquid, are separated into fractions of narrow b.p. range, each fraction being then subjected separately to destructive hydrogenation under pressure. If the initial material is solid, it is first converted wholly or partially, by some known process, into liquid products. Liquid starting materials may also be subjected to a preliminary hydrogenation, with or without the employment of pressure or catalysts, in order to modify their boiling ranges, before submitting them to the described process. A. B. MANNING.

Conversion of saturated hydrocarbons, in gas or vapour form, especially methane, into unsaturated hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 264,845, 19.1.27. Ger., 20.1.26).—The saturated hydrocarbons are passed through a disc-shaped flame of high temperature, formed by combustion of a by-passed portion of the gas with air or oxygen. Acetylene is the principal product. A. B. MANNING.

Cracking and hydrogenation of hydrocarbons, oils, or tars. P. WÜRTH (F.P. 622,715—6, 9.10.25).—A mixture of hydrogen sulphide and (A) the products obtained from a cracking process or (B) hydrocarbon vapours is subjected to a process in which nascent hydrogen is liberated by the thermal decomposition of the hydrogen sulphide. A. R. POWELL.

Treatment of oil-bearing shales and kindred oil-bearing minerals. N. H. FREEMAN (B.P. 291,475, 1.12.26; cf. B.P. 248,014; B., 1926, 396).—In cases where the preliminary heat-treatment of the shales etc. causes considerable liberation of liquid products, the latter are allowed to act as solvents and entraining agents in place of added solvent. Superheated steam or oil vapours may also be used as entraining and distilling agents. C. O. HARVEY.

Treatment of shale or the like. H. D. RYAN (U.S.P. 1,672,231, 5.6.28. Appl., 2.1.20. Renewed 23.8.27).—The shale is ground with a hydrocarbon oil at a temperature which, whilst sufficient to melt the bituminous material, limits the vaporisation, so that the mixture remains fluid. The bitumens are finally separated from the solid matter. F. G. CLARKE.

Purification of mineral oils. M. L. CHAPPELL, G. J. ZISER, and E. L. MOYER, ASSRS. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,672,304, 5.6.28. Appl., 18.8.24).—In the manufacture of lubricating oil,

the oil, after acid treatment and separation of the acid sludge, is repeatedly extracted with an alkali solution containing 90% by vol. of ethyl alcohol.

C. O. HARVEY.

Refining of [hydrocarbon] oil. J. R. NELLER and G. M. VANCE, ASSRS. to TEXAS CO. (U.S.P. 1,672,621, 5.6.28. Appl., 30.4.23).—Colour-forming constituents are removed from the acid-treated and neutralised oil by washing with alcoholic alkali solution.

C. O. HARVEY.

Purifying the used lubricating oil of internal-combustion engines. R. WISCHIN (B.P. 290,923, 8.12.27. Ger., 3.11.26).—The oil is mixed with concentrated sulphuric acid in a water-jacketed vessel and the temperature of the mixture is gradually raised (4–5°/hr.) to 50–60°. After separation of the sludge the oil is passed through a filtering agent, *e.g.*, fuller's earth, and is then treated with superheated steam at about 200°. A. B. MANNING.

Mineral lubricating, transformer, or other oils exposed at raised temperatures to air. F. HOFMANN and M. DUNKEL (B.P. 284,616, 22.11.26. Ger., 24.11.25).—Oxidation of the oils is inhibited by adding up to 2% of an acid amide, such as urea or oxamide, or a nitrile (cf. B.P. 262,107; B., 1928, 472).

C. O. HARVEY.

Liquid fuel. M. MÜLLER-CUNRADI, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,673,620, 12.6.28. Appl., 4.2.25. Ger., 4.2.24).—A liquefied mixture of butane and butylene is claimed. F. G. CROSSE.

Purifiers [with removable covers] for use in the manufacture of gas. WHESOE FOUNDRY & ENGINEERING CO., LTD., and R. L. CHAMBERS (B.P. 290,793, 1.4.27).

Apparatus for promoting the combustion of fuel. R. H. HEPBURN (B.P. 291,180, 22.3.27).

Washing of coal (U.S.P. 1,672,448).—See I. **Hydrogen from gas mixtures** (B.P. 291,576).—See VII.

III.—ORGANIC INTERMEDIATES.

Higher aldehydes. SCHIMMEL & Co. (Ber. Schimmel, 1927, 131; Chem. Zentr., 1927, II, 1518).—The aldehydes known in commerce as aldehydes C_{14} , C_{16} , C_{18} , and C_{20} belong, in their chemical nature, to ketones and esters, and they do not correspond in carbon content with their names. E. H. SHARLES.

"Nitrohumic" acid. FUCHS. **Action of sulphuric acid on aromatic hydrocarbons.** TILICHEV and DUMSKI.—See II.

PATENTS.

Manufacture of aromatic nitriles. I. V. GILES, ASSR. to AMER. CYANAMID CO. (U.S.P. 1,672,253, 5.6.28. Appl., 25.8.26).—Halogenated aromatic hydrocarbons are heated with copper cyanide in the presence of an alkali-forming metal cyanide. F. G. CLARKE.

n-Butyl lactate. C. L. GABRIEL and C. BOGIN, ASSRS. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,668,806, 8.5.28. Appl., 29.11.26. Renewed 24.2.28).—A mixture of 70% lactic acid and excess of *n*-butyl alcohol is distilled at 92° whereby water is removed as the constant-boiling mixture with butyl alcohol. The residual liquid

in the distillation apparatus is saturated with dry hydrogen chloride and refluxed for 1 hr. On cooling, the lower layer of *n*-butyl lactate (b.p. 186—189°) is separated, washed, dried, and distilled. A. R. POWELL.

***n*-Tributyl borate.** W. J. BANNISTER, Assr. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,668,797, 8.5.28. Appl., 26.9.27).—Boric acid is distilled with an excess of butyl alcohol until the temperature rises suddenly from 117° to 150°, the excess of butyl alcohol is then distilled off in a vacuum, and the residual *n*-butyl borate, b.p. 190°/220 mm., is purified by distillation under reduced pressure. A. R. POWELL.

IV.—DYESTUFFS.

Alizarinsulphonic acid ester—a water-soluble alizarin preparation. E. GEBAUER-FÜLNEGG and I. EISNER (Ind. Eng. Chem., 1928, 20, 637—638).—With chlorosulphonic acid in pyridine alizarin affords the *monopyridine salt* of an alizarinmonosulphonic acid, $C_{19}H_{13}O_7NS$, which when treated with calcium or barium hydroxide or with the alkali carbonates affords the corresponding bimetallic salts, $C_{14}H_6O_2(OM) \cdot OSO_3M$. In dimethylaniline lower yields are obtained and fractional crystallisation is necessary to separate the sodium salt from sodium acetate. All salts of the ester undergo quantitative hydrolysis with concentrated hydrochloric acid. The pyridine salt is decomposed on heating at 110° and alizarin is regenerated on crystallising this salt from boiling water. Brilliant and clear shades of alizarin-red can be obtained by padding material, previously treated in the usual manner with aluminium and calcium salts, with a 1—4% solution of this pyridine salt (containing 2.5% of pyridine for each 1% of dye). After drying, the orange-yellow material is heated at 2 atm. in a Mather-Platt for 10—15 min. and washed with hot soapy water to remove non-hydrolysed and insufficiently fixed calcium and aluminium salts of the alizarin ester, the latter salts requiring the presence of an acid for decomposition at this temperature. Better results are obtained by padding with a cold solution containing 3—5% of the sodium or potassium salt of the alizarinmonosulphonic acid ester and an equal amount of oxalic acid, and, after drying, developing in the Mather-Platt at 110° for 10—15 min. with fairly dry steam; after-washing is unnecessary. R. BRIGHTMAN.

PATENTS.

Manufacture of water-soluble ester-like derivatives of vat dyes. W. BAUER, Assr. to DURAND & HUGUENIN Soc. ANON. (U.S.P. 1,668,392, 1.5.28. Appl., 15.10.26. Ger., 26.10.25).—Leuco-compounds of indigoid vat-dyes (e.g., dibromoindigo-white) are treated with pyrosulphuryl chloride in the presence of an organic base, e.g., dimethylaniline (cf. U.S.P. 1,448,251; B., 1923, 398 A). A. R. POWELL.

Manufacture of vat dyes of the 2-thionaphthen-2'-indoleindigo series. I. G. FARBENIND. A.-G. (B.P. 266,582, 21.2.27. Ger., 20.2.26. Cf. B.P. 274,527; B., 1927, 743).—Vat dyes especially fast to boiling sodium carbonate solution are obtained by condensing thioindoxyls, carrying in positions 4, 6, and 7 three halogen atoms, or two halogens and an alkyl group, or one halogen and two alkyl groups, with an isatin- α -anil or

α -halide. Thus, 6 : 7-dichloro-4-methylthioindoxyl, prepared from 4-chloro-*o*-toluidine by way of 5 : 6-dichloro-2-amino-3-methylphenylthioglycollic acid (*lactam*, m.p. 231—232°) gives with 5 : 7-dichloroisatin α -chloride a reddish-violet vat dye. C. HOLLINS.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Dispersoidological investigations. XVII. Application in industry of silk-fibroin, and other similar chemically-composite compounds, in its colloidal solutions. P. P. VON WEIMARN (Rep. Imp. Ind. Res. Inst. Osaka, 1928, 8, 47 pp.; cf. B., 1912, 768).—Threads produced under suitable conditions by coagulating concentrated solutions of silk in neutral solutions of neutral salts (e.g., in sodium iodide or thiocyanate solutions) by means of an aggregating solution (sodium citrate or sodium potassium tartrate) remain unchanged for more than a year. Under less favourable conditions of preparation they become brittle. Ultramicroscopical investigation of the phenomenon of "fibril cleavage" in the swelling of natural silk fibres shows that the latter can be divided into fibrils smaller than 200 $\mu\mu$ in size, i.e., they are of colloidal dimensions in thickness and breadth. Threads obtained from a silk-tannin coagulum are more stable the greater the elimination of tannin from them. Vegetable or wool fibres coated with silk fibroin (e.g., by immersion first in a 35—40% solution of silk in sodium iodide solution and then in a coagulating bath of sodium potassium tartrate) become more lustrous and stronger, and the increase in strength of woollen fibres so treated is very pronounced. Scroop and crêpon effects are produced by treatment of silk material first with a neutral salt swelling agent such as sodium iodide, and then with a coagulating agent, there being less likelihood of damage to the fabric than when concentrated acids are used for a like purpose. Concentrated aqueous salt solutions of such composite substances as casein, chitin, keratin, and fibrin are suggested for use in the textile industry (cf. A., 1926, 1203) and an investigation of the action of very soluble salts corresponding with the Hofmeister series on textile fibres is recommended. B. P. RIDGE.

Natural silk. I. Iodine-absorbing power of silk fibroin. T. TAKAHASHI (J. Soc. Chem. Ind. Japan, 1928, 31, 152—155).—Silk fibroin, which had been treated with various reagents and regenerated, showed varying iodine-absorbing power, thus indicating that the chemical reactivity of fibroin depends greatly on its fibrous structure. Y. TOMODA.

Crystalline-liquid state as a general property of matter. VI. Aggregative liquid-crystal state of natural silk. P. P. VON WEIMARN (Kolloid-Z., 1928, 45, 161—162).—A description is given of the microscopic structures produced by pouring a concentrated solution of silk in a dispersing medium into a coagulating solution. These forms are compared with those produced in the precipitation of barium sulphate (A., 1928, 584).

E. S. HEDGES.

Degumming of silk. V. Influence of fatty acid. T. TAKAHASHI (J. Soc. Chem. Ind. Japan, 1928, 31, 148—151).—Fatty acid prevents the degumming of silk either in acid or in alkaline medium, but the effect

is not distinct when the acidity of the medium is above the isoelectric point of sericin. It appears that the degumming action of soap solution is due to its alkalinity, whereby the sericin becomes dissolved and the fatty acid or acid soap liberated as the result of combination of alkali with sericin is adsorbed on the fibre and protects the fibre from the further action of alkali.

Y. TOMODA.

Loss of strength [tendering] of cotton exposed to light. P. W. CUNLIFFE and F. D. FARROW. **Effect of structure and of bleaching on the strength of cotton yarns.** P. W. CUNLIFFE and E. MIDGLEY (J. Text. Inst., 1928, 19, 169—188 T).—Comparison is made between the strength of cotton in the form of hairs, yarn, or cloth after exposure to mercury-vapour lamps, the carbon arc, and to sunlight, both in England and abroad, and that of similar unexposed materials. Cotton hairs exposed to the quartz mercury-vapour lamp lose strength at a constant rate until the tendering amounts to about 40%; the rate then diminishes. Over a small range the rate of tendering is proportional to the intensity of the light. There are specific differences between the susceptibilities to light tendering of cotton hairs of different origin, and destruction proceeds more rapidly in the presence of oxygen, water, or carbon dioxide than in hydrogen, nitrogen, or *in vacuo*. The proportional loss of strength of yarns is about half that of hairs fully exposed under the same conditions, the hairs in the yarns being tendered to the same extent as the yarns themselves. Fine yarns are more rapidly tendered than coarse, and soft twisted than hard, both for bleached and raw cotton. Bleached cotton is more rapidly tendered than raw, and material dyed chrome-green is much more resistant to destruction than the undyed, scoured material. Ferric hydroxide has a slight protective action, whilst cloth dyed mineral-khaki is as resistant as the chrome-dyed material, the effect being such as to extend the life of fabric subject to strong illumination to as much as five or six times that of untreated material. Organic dyes modify the rate of tendering of cotton by light, some unfavourably, others favourably, the effects never being so large as those found with chromium hydroxide. In an appendix it is observed that when the results of breaking tests on yarns of different counts are made comparable by expressing them in terms of the strength of equal weights of cotton in a standard length, for the same twist constant, coarse yarns are inherently stronger than fine, and doubled yarns are stronger than the two singles from which they are spun, but weaker than a single yarn equal in actual counts to the doubled. Bleached yarns are weaker than raw, and, taking the work done in breaking a standard strip of cloth as a measure of its strength, bleached cloth is weaker than unbleached.

B. P. RIDGE.

Relation between atmospheric humidity and breaking strengths and extensibilities of textile fabrics before and after weathering. A. J. TURNER (J. Text. Inst., 1928, 19, 101—168 T).—Doped and undoped flax and cotton fabrics, ramie and jute fabrics, brown paper, cellophane sheet, cellulose acetate film, and pigmented nitrocellulose varnish film were tested in an

Avery machine at various relative humidities from 30% to saturation, before and after weathering for different periods. The warp of cotton fabrics is more affected by humidity changes than the weft, and so are coarse than fine fabrics, whilst mercerised cotton fabrics are less sensitive to such changes than unmercerised. Physical differences, such as differences in the counts and twist of yarns from the same fibre, and the calendering of fabrics, do not appreciably alter the "humidity-strength coefficient" of the material (*i.e.*, the percentage increase of strength for a 1% increase of humidity expressed in terms of the strength at 70% R.H.), but chemical treatments such as the mercerisation of cotton, or the cuprammonium or bitumen proofing of flax fabrics, cause considerable changes in this factor. The effect of doping depends largely on the extensibility of the material treated, whilst the effect of humidity on doped fabric corresponds roughly to the sum of its separate effects on the fabric and on the dope. The humidity-strength coefficients of all classes of fabrics are greatly changed as a result of weathering, the strength and extensibility of the weathered fabric decreasing with increasing humidity.

B. P. RIDGE.

Pentosans in the sulphite[-cellulose] cooking process. L. P. ZHEREBOV (Bumazhnaya Promyshlennost, 1927, 6, 387—404).—When heated with sulphite cooking acid above 80° (aspen) pentosans form a compound with calcium hydrogen sulphite in which 1 mol. is attached to a pentosan molecule consisting of four pentose remnants; at 136° the ratio is 1 mol. to 2 pentose remnants, and hence the basic pentosan unit is regarded as $(C_5H_8O_4)_2$. Above 136°, disintegration of the molecule takes place with production of sulphuric acid or sulphate and carbon. On cooking with hydrogen sulphite the compounds resulting from the decomposition of the pentoses contain 1 mol. of sulphur dioxide for each reacting pentose molecule. The action of hydrogen sulphite differs from that of free sulphur dioxide. Replacement of calcium by magnesium results in less decomposition of pentosans. The unstable compound which is decomposed by ammonia, liberating calcium sulphite, results from interaction of the hydrogen sulphite with lignin or hexosans, but not with pentosans.

CHEMICAL ABSTRACTS.

Alkali-soluble constituents of sulphite-celluloses and artificial silks. W. WELTZEN (PapierFabr., 1928, 26, Fest-u. Auslands-Heft, 115—120).—The solubility of different kinds of cellulose and of artificial silk in sodium hydroxide solutions at 18—19° has been determined. On plotting the percentage of cellulose dissolved against concentration of sodium hydroxide solutions (in vol.-%) characteristic curves are obtained which in all cases rise steeply to a maximum corresponding to 10—12% of sodium hydroxide. Increasing the time of treatment of the cellulose from 3 to 30 hrs. gives maximum solubility in a solution containing nearer 10% than 12% of sodium hydroxide. The percentage of cellulose dissolved under these conditions of maximum solubility varies with the material examined, 15—25% of sulphite cellulose, 30% of cuprammonium silk, 40—50% of viscose, and 100% of nitro-silk being representative values.

B. P. RIDGE

Washing and treatment of sulphite-cellulose during its preparation. D. E. OMAN (Papier-Fabr., 1928, 26, Fest- u. Auslands-Heft, 92—96).—The properties of paper produced from sulphite-cellulose are to some extent dependent on the p_H value of the liquid with which the latter is washed. For some papers, change of this value to the alkaline side, *e.g.*, by the addition of milk of lime, gives better sizing properties and an improved appearance of the final material. For grease-proof paper, on the other hand, washing with a slightly acid liquid appears advantageous.

B. P. RIDGE.

Determination of the degree of decomposition of sulphite-celluloses. E. HÄGGLUND (Papier-Fabr., 1928, 26, Fest- u. Auslands-Heft, 88—92).—The lignin content of non-bleachable sulphite-cellulose, which represents the true degree of decomposition, is proportional to the chlorine consumption as determined by the methods of Enso and Sieber, and to the bromine absorption as determined by Tingle's method. The Roschier numbers, and values given by other permanganate methods (*e.g.*, Björkman's), correspond approximately with the degree of decomposition only for very hard celluloses with a greater lignin content than 3—4%; for a greater degree of decomposition, *i.e.*, a lower lignin content, there is no proportionality, the deviation being due to the greater or less degree of sulphonation of the lignin—the more highly sulphonated lignin being less easily oxidised. In general, no relation holds between the degree of decomposition and the strength of the fibre, although if equal cooking conditions be assumed, it is possible to specify exact relations between these factors.

B. P. RIDGE.

Action of strong sodium hydroxide on cellulose. P. WAENTIG (Papier-Fabr., 1928, 26, Fest- u. Auslands-Heft, 64—66; cf. B., 1927, 773).—The spontaneous decomposition of alkali-cellulose at the ordinary temperature is caused by the action of oxygen from the air, which is absorbed by the material. It is concluded that the oxycellulose content of the alkali-cellulose is not thereby increased, since, although its α -cellulose content is considerably decreased as a result of prolonged exposure to the air in contact with sodium hydroxide solution, its copper number is not significantly raised. Alkali-cellulose from American cotton is more resistant to decomposition under these conditions than that from pinewood sulphite-cellulose. If air be completely excluded, no decomposition takes place; on the contrary, the α -cellulose content of the material is slightly increased, whilst the corresponding viscose solutions obtained from it are more viscous.

B. P. RIDGE.

Quality control in the sulphite pulp industry. A. LAMPÉN (Zellstoff u. Papier, 1928, 8, 289—293).—The factors which affect the quality of sulphite pulp are outlined and discussed. The chief cause of variations in quality is lack of uniformity in the pulp wood itself as regards age, rapidity of growth, and moisture content. The various methods of testing sulphite pulp are described, and a plea is advanced for the introduction of standard methods to avoid the present confusion. Bleach consumption is most accurately indicated by the Bergman chlorine number, which corresponds very

closely with the actual chlorine consumption of the pulp on the large scale. For the determination of strength after beating, a single-ball mill is recommended; the most reliable results are obtained if the pulp is always beaten to the same degree, *viz.*, 75° as measured by the Schopper-Riegler tester. At 300 r.p.m., the time required varies from 1½ to 2½ hrs., and in this way useful indications are obtained of the beating properties of the pulp. The strength of the final test sheet is further influenced by the pressure exerted during the formation of the sheet, the dilution of the pulp in the sheet mould, the intensity of suction, the weight of the sheet, and its moisture content. Each of these factors would require to be rigidly defined in any proposed standard method for determining the strength of pulps.

D. J. NORMAN.

Rosin sizing [of paper]. S. R. H. EDGE (World's Paper Trade Rev., 1928, 89, 616—620).—The time of penetration in the ferric chloride-ammonium thiocyanate sizing test is correlated with the bulk of the sheet and the temperature of drying. Sizing is not proportional to the rosin content of the paper. CHEMICAL ABSTRACTS.

Detection of sulphur and sulphurous acid. NOLL.—See VII.

PATENTS.

Treatment of natural silk. S. S. SADTLER and E. C. LATHROP (U.S.P. 1,671,786, 29.5.28. Appl. 1.6.25).—The partially spent liquor which has been used for softening one batch of silk is made up to its original volume with fresh softening liquor, and is then treated with an alkaline solution in sufficient quantity to adjust the p_H of the bath to such a value that a further batch of silk may be softened therein without injury.

D. J. NORMAN.

Treatment of wool. J. A. CRAVEN, and YORKSHIRE DYEWARE & CHEM. CO., LTD. (B.P. 290,805, 23.4. and 18.5.27).—The shrinkage of wool, caused by washing or milling in alkaline soap solutions, is considerably reduced by precipitating basic salts of iron, aluminium, or chromium on the fibre, or by treating it with a slightly alkaline solution of formaldehyde. *E.g.*, the material may be soaked for about 24 hrs. in a dilute basic solution of iron ammonium alum, after which it is rinsed and neutralised with borax or other mild alkali. If desired, a final treatment with a soap-oil emulsion or a sulphonated oil may be given. With chromium compounds a single or two-bath chrome tanning process may be adopted, but in the latter case it is preferable to use sodium bisulphite as the reducing agent. The wool may in all cases be pretreated with alkalis and then, if desired, with a natural or artificial bate.

D. J. NORMAN.

Manufacture of artificial silk etc. by the dry or evaporative method. N. V. NEDERLANDSCHE KUNST-ZIJDEFABR. (B.P. 282,326, 12.1.27. Holl., 17.12.26).—The solution is spun downward into a cylindrical spinning chamber (about 2 m. long and 25 cm. in diam.) open at the bottom and provided with a jacket, the circulation of the heating medium through this jacket being so regulated that the temperature gradually falls, *e.g.*, for a solution of cellulose acetate in acetone, from 50° at the top to 20° at the bottom. The air or inert gas into

which the filaments are spun flows slowly, *e.g.*, at about 8 cm./sec., upward through the chamber, and is withdrawn through an adjustable outlet near the spinning nozzle. In this way turbulence of the evaporative medium is practically eliminated, and, during the earlier stages of filament formation, sufficient solvent vapour is present to ensure the thread having a uniform cross-section.

D. J. NORMAN.

Manufacture of artificial threads, filaments, bands, etc. COURTAULDS, LTD., and [A] W. H. STOKES, [B] C. F. TOPHAM, E. HAZELEY, and E. A. MORTON (B.P. 290,693 and 290,833, [A] 15.1. and 5.11.27, [B] 23.5.27).—(A) Substances such as oils, waxes, finely-divided solids, leuco-compounds of vat dyes, etc. which it is desired to incorporate with viscose threads are fed in predetermined quantity, optionally in the form of a concentrated solution or suspension in a small quantity of the spinning solution, into the filtered and de-aerated viscose solution on its way to the spinning nozzles. Efficient mixing is ensured by the provision of a series of finely-perforated plates supplemented by a small mixing chamber containing a rotating paddle. Air should be excluded at all stages of the mixing operation. (B) When artificial silk produced by box-spinning processes is washed and dried in cake form, an uneven thread results owing to the fact that the threads on the outside of the cake dry under a greater tension than that to which the inside threads are subjected. This may be avoided by deforming the annular cake before drying, by, *e.g.*, pressing the cake inwards at four approximately equidistant points on its circumference to such an extent that the thread at all parts dries without undue tension.

D. J. NORMAN.

Manufacture of cellulose acetate. H. LE B. GRAY and C. J. STAUD, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,668,959, 8.5.28. Appl., 17.2.26).—Clear, grainless cellulose acetate is produced from cheap cellulose material, such as cotton linters, cotton dust, and sulphite pulp, by first treating it at 20–30° with 35–75% sulphuric acid, whereby injurious anhydride constituents are removed without degrading the cellulose. After washing until free from acid, and drying, the acetylation is performed using zinc chloride, or chlorine and red phosphorus, or other condensing agent less active than sulphuric acid.

F. G. CLARKE.

Removal of impurities from paper pulp and the like. S. MILNE (B.P. 291,154, 25.2.27).—The pulp is suspended in water and passed through a series of vessels of such design, *e.g.*, containing a central partition extending almost to the bottom, that the mixture flows therethrough in a tortuous up-and-down path. The impurities settle to the bottom and are continuously withdrawn through a grating by a pump, which returns them into the top part of the preceding vessel in the series until ultimately they are collected in a strainer above the main water supply tank.

D. J. NORMAN.

Treatment of cellulosic fabrics. ARNOLD PRINT WORKS, Asses. of D. DEGÖNCZ and A. S. JONES (B.P. 282,002, 3.9.27. U.S., 9.12.26).—See U.S.P. 1,661,881; B., 1928, 330.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Effect of adding oxidants in the steeping process [in bleaching]. HALLER and P. SEIDEL (Z. angew. Chem., 1928, 41, 698–702).—Contrary to earlier views it is shown that the addition of oxidising agents at the steeping stage in the process of bleaching is undoubtedly advantageous. Oxidising agents, however, which readily give up their oxygen in the alkaline bath are to be avoided; the best results are obtained with "aktivin" (sodium *p*-toluenesulphonchloroamide), which affords on hydrolysis neutral easily soluble products removable by washing. The use of such agents enables the bleaching to be conducted at lower pressures, and a steeping period of 4 hrs. serves instead of 6–8 hrs. as formerly. Satisfactory results are not obtained by steeping with lime alone; a further treatment with 2% sodium hydroxide solution gives a well-bleached product. In the steeping process the loss in weight is proportional to the alkali consumed and the organic material dissolved, and the function of the oxidising agent appears to be that of converting the impurities into products soluble in alkali. Microscopical examination shows that the operation removes pectins and the cuticle incrustation without impairing the tensile strength of the material. Inferior results with lime alone appear to be due to the failure of this reagent to act on the cuticle. Oxycellulose is only formed to a slight extent as shown by the copper numbers and "boiling-out" numbers of the steeped material. The best results are obtained with a bath containing about 10% of aktivin (corresponding to 2% of "active" chlorine) and 2% sodium hydroxide solution. For the tests, crude cellulose from nettles, which gave no starch reaction with iodine, was used.

R. BRIGHTMAN.

Bleaching of sulphite-cellulose in higher stuff-density. E. HOCHBERGER (Papier-Fabr., 1928, 26, Fest-u. Auslands-Heft, 66–88).—Bleaching in higher stuff-density has some advantages especially in the prebleaching, the precise conditions for which have been investigated. For the prebleaching of a readily bleached cellulose the variable factors involved change according to simple laws; thus, the rate of bleaching is proportional to the initial concentration of the bleaching agent, and the reaction velocity to the square of the concentration of unchanged bleaching agent at the time considered. Dilution of the active bleaching agent increases in arithmetical progression, whilst the p_H value decreases rectilinearly with time. The efficiency of the bleaching process, especially during the after-bleach, depends on the velocity of diffusion of the soluble incrusting substances from the interior of the fibre into the surrounding liquid, and thorough washing between the pre- and after-bleaching is necessary not only to increase this velocity, but also to remove the incrusting substances in the liquor which would otherwise use up chlorine unnecessarily.

B. P. RIDGE.

Determination of chlorate-chlorine in bleach liquor. H. PRELINGER (Zellstoff-u. Papier, 1928, 8, 294–295). The ferrous sulphate-permanganate method for determining chlorates is inadmissible in the case of spent bleach liquor owing to the presence therein of

oxidisable organic matter. Satisfactory results may, however, be obtained by determining the chloride-chlorine by Volhard's method before and after reduction with sulphurous acid. Active chlorine, if present, should first be quantitatively converted into chloride by alkaline hydrogen peroxide solution. D. J. NORMAN.

Causes of uneven dyeing [of wool]. H. R. HIRST (J. Soc. Dyers and Col., 1928, 44, 163—169).—Curves showing the rates of absorption at various temperatures of dyes by wool are given, and it is shown possible by means of them to select conditions of dyeing favourable to the production of level shades. It is suggested that dye-makers should issue similar curves for all wool dyes. The presence of unequally distributed alkali in scoured wool promotes uneven dyeing afterwards. *E.g.*, wool immersed for 20 min. in a solution of sodium carbonate (*d* 1.026), whereby it retained 1.14% Na_2CO_3 , had an increased affinity for dyes, whilst wool similarly treated with a solution of sodium carbonate (*d* 1.050), whereby it retained 1.42% Na_2CO_3 , had a decreased affinity. Wool treated with an alkaline solution and then thoroughly washed dyes to a fuller shade than similarly treated wool only slightly rinsed. Wool impregnated with a dilute alkaline solution and dried has a decreased affinity for acid dyes, but if steamed instead of being dried its affinity is considerably increased. Thus, wool impregnated with a solution of sodium carbonate (*d* 1.006) and then steamed for 7.5 min. gave a dyeing 48% fuller in shade than dyeings obtained on wool similarly treated with an alkali but not steamed. The affinity of wool for indigo, acid, and chrome dyes is decreased by bacterial attack. Neutral or alkaline wool is particularly susceptible to attack by spore-forming bacteria, such as are present in the soil, *e.g.*, *B. mesentericus* and *B. subtilis*; the optimum moisture content of wool favourable to bacterial attack is 30%.

A. J. HALL.

Reserve salts [in textile printing]. C. M. KEY-WORTH (J. Soc. Dyers and Col., 1928, 44, 177—178).—A number of reserve salts have been identified by means of their arylamine salts (A., 1927, 235) prepared by treating 5 g. of the commercial reserve salt dissolved in 50 c.c. of water with 2.5 g. of the hydrochlorides of aniline, or *o*- or *p*-toluidine dissolved in 20 c.c. of water, and crystallising the product from 0.2*N*-acetic acid. They consisted in most cases of sodium nitrobenzene-*m*-sulphonate; Reserve Salt O (Leon) was identified as sodium nitrotoluene-*p*-sulphonate.

A. J. HALL.

Improvements in dry-cleaning and laundry processes. A. E. HATFIELD and E. A. ALLIOTT (J. Soc. Dyers and Col., 1928, 44, 170—177).—A new cyclic dry-cleaning process is described in which soiled garments are treated with a solvent-soap solution in a rotating washing machine, the soiled liquor is rapidly and continuously withdrawn, pumped through a Sweetland filter press after the addition of a filter acid, and then returned to the washing machine without any intermediate process of settling or distillation. Advantages over other methods are absence of re-settlement of dirt on the cleaned articles, small losses of solvent and soap, and the articles are practically freed from bacteria, these being retained by the filter-press cake.

A. J. HALL.

Fastness of dyed fabrics to laundering. R. G. PARKER and D. N. JACKMAN (J. Text. Inst., 1928, 19, 223—232 T).—Methods hitherto suggested for testing fastness to washing are criticised and the necessary treatments for different classes of goods during laundry washing processes summarised. It is suggested that in any system of testing the fastness of dyes the following should be taken into account: the purpose for which the dyed fabric may be used; the vigour of the cleansing process necessary to remove stains; the effect of the mechanical motion of a relatively large weight of fabrics in a rotary washing machine, also of repeated laundering, use, exposure to light, moisture, fog, and perspiration; and effects due to textile finishing processes on the subsequent washing properties of the fabric.

B. P. RIDGE.

Alizarinsulphonic acid ester. GEBAUER-FÜLNEGG and EISNER.—See IV.

PATENT.

Treatment of penetrable [fibrous] substances by liquids. COHOE PROCESSES INC., Assees. of W. P. COHOE (B.P. 263,113, 16.11.26. U.S., 19.12.25).—Liquid, such as dye, is forced through material, such as cloth, by submerged rollers running at a speed of the order of 1,500 ft./min. in close proximity to the cloth, the layer of liquid dragged by the skin friction of the roller being sufficient to prevent actual rubbing between the roll and the cloth which must move at very different speeds if not in opposite directions. Various arrangements of rolls are described.

B. M. VENABLES.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Ammonia oxidation replaces nitre for chamber acid plants. S. F. SPANGLER (Chem. Met. Eng., 1928, 35, 342—344).—The use of ammonia oxidation plants in connexion with the lead-chamber sulphuric acid process is rapidly extending in America. The stripping of the 25% ammonia solution used is now effected without steam by the waste heat from the converter. Preheating of the ammonia-air mixture is also usual, but it must be done in an aluminium or nickel pipe owing to the catalytic effect of iron. Cylindrical gauzes are most usual, and the converter chamber is lined with firebrick. It is customary to control the temperature of the converted gases with a thermocouple. The relative proportions of ammonia and air used may be automatically controlled.

C. IRWIN.

Volumetric and thermal study of ammonia and its synthesis. R. NITZSCHMANN (Chem. Listy, 1928, 22, 169—177, 199—202, 241—244).—The relations between pressure, temperature, and composition of mixtures of nitrogen and hydrogen are determined, and, together with thermal exchanges and liquefaction after production of ammonia, are expressed in a number of equations and graphs, whence may be calculated, amongst other data, the optimum dimensions for the surface of exchange under given conditions. The values obtained for the calorific exchange are of a very high order.

R. TRUSZKOWSKI.

Ammonia liberation from ammonium sulphate solutions by various limes. R. A. RACATZ and

J. M. SPEES (Gas Age Rec., 1927, 60, 953—955).—The presence of magnesia in lime used for the liberation of ammonia from ammonium sulphate solutions does not affect the total yield in 90 min., but diminishes the rate of evolution in the early stages.

CHEMICAL ABSTRACTS.

Direct gypsum process of ammonium sulphate recovery. H. W. JACKMAN (Gas Age Rec., 1927, 60, 107—108, 116).—In the process for the production, on a semi-commercial scale, of ammonium sulphate by passing ammonia and carbon dioxide through water containing gypsum, 52.4% of the ammonia was recovered as sulphate.

CHEMICAL ABSTRACTS.

Indirect gypsum process for sulphate manufacture. R. A. HOFFMAN (Gas Age Rec., 1928, 61, 111).—In the production of ammonium sulphate from gas liquors and gypsum, the average yield was 65% and the purity of the product 95%. Discoloration was due to the action of hydrogen sulphide on the iron evaporator.

CHEMICAL ABSTRACTS.

Analysis of sodium sulphate used in glass manufacture; direct determination of Na_2SO_4 . A. BESOMBE (Bull. Soc. chim. Belg., 1928, 37, 164—165).—A sample (2 g.) is dissolved in 30 c.c. of water and the carbonates of iron, aluminium, calcium, and magnesium are precipitated by adding excess of 0.1*N*-sodium carbonate. The solution is made up to 100 c.c., filtered, and 50 c.c. of the filtrate are placed in a tared basin and exactly neutralised to methyl-orange with 0.2*N*-sulphuric acid. The solution is evaporated to dryness, heated to dull redness for a few moments, cooled, and weighed. The sodium chloride present and the sodium sulphate added are subtracted from the weight of the residue to give the weight of sodium sulphate originally present in 1 g. of the sample.

W. J. BOYD.

Stable sodium thiosulphate solution. L. W. WINKLER (Pharm. Zentr., 1928, 69, 369—371).—Decomposition of 0.01*N*-sodium thiosulphate solution by exposure and by sulphur bacteria can be prevented by the addition of 0.01% of mercuric cyanide. A solution thus prepared remained clear and had a constant titre over a period of two months, whereas an unpreserved solution decreased in thiosulphate content and deposited sulphur. Details of the preparation of sodium thiosulphate solution and its standardisation both with potassium iodate and iodine are given. For most accurate work the thiosulphate should be standardised against pure, sublimed iodine, the preparation of which is described.

E. H. SHARPLES.

Manufacture of barium and strontium nitrates. F. CHEMNITZ (Chem.-Ztg., 1928, 52, 465—466).—An account of the production of barium nitrate from the carbonate and of strontium nitrate from the hydroxide.

Sensibility of the thermobalance and its use in the analysis of hydrated lime. T. SOMIYA (J. Soc. Chem. Ind. Japan, 1928, 31, 217—223).—A modification of Honda's thermobalance is described; in accuracy it is claimed to be equal to that of the analytical balance even at temperatures above 1000°. By its means a sample of hydrated lime can be quantitatively analysed. The sample, suspended from one end of the beam in

an electric furnace, is weighed continuously, and curves relating temperature to loss in weight are plotted. The percentage of hydroxide present was calculated from the amount of water given off at 400—500° and that of carbonate from the amount of carbon dioxide evolved at 500—950°. The amount of calcium hydroxide as determined by this method was less than the theoretical amount by 2.1%, but that of calcium carbonate agreed.

Y. TOMODA.

Chemical engineering may revive sublimation of sulphur. E. F. WHITE (Chem. Met. Eng., 1928, 35, 355—356).—Although the competition of ground sulphur has rendered the older forms of subliming plant uneconomic, yet flowers of sulphur are superior to the ground product for both vulcanisation and agricultural purposes. The author employs protected sheet iron in place of brick for condensing chambers and an arrangement of condensing surfaces. The firing is by a separate furnace, and the inert atmosphere is of flue gases instead of sulphur dioxide, which are circulated with a fan. Entrained molten sulphur is separated by a baffle, and the flowers of sulphur are removed mechanically from the chambers without stopping the plant. It is claimed that with this plant costs are very little higher than with grinding.

C. IRWIN.

Detection of sulphur and sulphurous acid. A. NOLL (Papier-Fabr., 1928, 26, Fest-u. Auslands-Heft, 59—61).—Sulphur (elementary or in combination) is detected by heating a sample of the substance with excess of sulphur-free iron powder, dissolving the melt in pure dilute hydrochloric acid, and demonstrating the presence of hydrogen sulphide by its odor or by means of lead acetate paper. Alternatively, the acid solution is rendered alkaline and the presence of sulphur shown by means of sodium nitroprusside solution. In testing for sulphurous acid in such materials as half-stuff or other products of the cellulose and paper industries, a sample of the material is pulped with distilled water, digested on the water-bath, filtered, acidified with hydrochloric acid, and chemically pure zinc added. The resulting hydrogen sulphide is recognised as above. Alternatively, the aqueous extract of the material is heated on the water-bath with zinc powder, whereby the sulphurous acid is converted into hyposulphite, the presence of which is shown by its characteristic reducing action upon solutions or test papers made from indanthrene-yellow G, methylene-blue, or the sodium salt of anthraquinone- β -sulphonic acid. When the latter are used it is not advisable to heat during the extraction of the material with water, since substances of a reducing nature other than sulphurous acid may thus be extracted, which would give the same colour changes with the dye solutions as hyposulphite.

B. P. RIDGE.

Rate of absorption of sulphurous gases by magnesium hydroxide [and sulphite]. P. RIOU and A. P. BÉBARD (Compt. rend., 1928, 186, 1465—1467; cf. B., 1928, 482).—The rates of absorption of the gases by saturated solutions of magnesium hydroxide or sulphite are similar to that by water, but increase in the presence of an excess of the solid

constituent. In both cases they decrease with rise in temperature.

J. GRANT.

Compounds and combustion. THOMAS and HOCHWALT.—See II. **Chlorate in bleach liquor.** PRELINGER.—See VI.

PATENTS.

[Catalyst for] contact sulphuric acid process. MONSANTO CHEM. WORKS, ASSEES. of A. O. JAEGER and J. A. BERTSCH (B.P. 266,007, 14.2.27. U.S., 15.2.26).—The catalyst comprises an artificial zeolite containing vanadium pentoxide in a non-exchangeable form. It may be mixed with suitable porous non-reactive carriers, or finely-divided porous materials, such as kieselguhr or asbestos, may be incorporated with it prior to drying. The prepared catalytic mass may be treated with a solution of one or more catalytically active compounds which enter into base exchange with the zeolite.

A. R. POWELL.

Containers for hydrofluoric acid and other highly corrosive chemicals. H. O. TRAUN (DR. H. TRAUN & SÖHNE) (B.P. 283,868, 10.8.27. Ger., 18.1.27).—The containers are constructed of artificial resins, preferably phenol-formaldehyde condensation products mixed with 15–25% of powdered graphite, and may be coated externally with varnish etc.

L. A. COLES.

Production of ammonium nitrate free from nitrite. ELEKTIZITÄTSWERK LONZA (GAMPEL & BASEL), and E. LÜSCHER (Swiss P. 118,962, 23.10.25).—Nitric oxide obtained, e.g., by the oxidation of ammonia, is passed together with oxygen into a saturated ammonium nitrate solution containing manganese compounds, and ammonia is added continuously or intermittently to neutralise the nitric acid or to interact with the manganese nitrate formed. The ammonium nitrate crystallising out is removed from the solution.

L. A. COLES.

Manufacture of alkali cyanides. C. P. DAVIS and G. B. FROIT, ASSRS. to AMER. CYANAMID CO. (U.S.P. 1,672,449, 5.6.28. Appl., 24.12.24).—Alkali cyanides are obtained by heating the corresponding carbonates in a current of hydrogen cyanide to progressively rising temperatures below the m.p. of the resulting mixture but sufficiently high to cause conversion of the greater portion of the carbonate into cyanide.

A. R. POWELL.

Preparation of [anhydrous] metal halides. J. W. MARDEN, ASSR. to WESTINGHOUSE LAMP CO. (U.S.P. 1,665,634, 10.4.28. Appl., 3.9.21).—Anhydrous halides of metals may be recovered from their aqueous solutions by evaporating the solutions to dryness after addition of an excess of the corresponding ammonium halide, and heating the dry residue in a current of the dry hydrogen halide free from oxygen. This procedure avoids hydrolysis of the metal halide.

A. R. POWELL.

Manufacture of alkali sulphhydrates [hydrogen sulphides]. H. P. BASSETT (U.S.P. 1,662,735, 13.3.28. Appl., 15.12.25).—A portion of the sodium sulphide content of a saturated solution of this compound is crystallised out, and the mother-liquor is treated with sodium hydrogen sulphate to obtain hydrogen sulphide, which is then caused to react with a solution of the crystals first obtained.

A. R. POWELL.

Production of a solution of cyanamide from calcium cyanamide. A. N. ERICKSON, ASSR. to UNION CARBIDE CO. (U.S.P. 1,671,183, 29.5.28. Appl., 20.4.27).—The calcium cyanamide is treated with carbon dioxide in the presence of water at 10–20°, whereby the carbonated lime-nitrogen compounds are precipitated and the production of dicyanodiamide is avoided. After partial removal of water the sludge is again treated with carbon dioxide at 32° and the calcium carbonate separated from the resulting solution of cyanamide.

F. G. CLARKE.

Preparation of titanium compounds and pigments. P. SPENCE & SONS, LTD., and S. F. W. CRUNDALL (B.P. 290,683—4, 17.11.26).—(A) Titanium phosphate is prepared by addition of a soluble phosphate to a solution of a titanium salt containing more than 10% of titania and more than 15% of free sulphuric acid or its equivalent. To obtain a precipitate that filters readily a temperature of 50° must not be exceeded. (B) Titanium phosphate is converted into hydroxide or oxide by heating with alkali carbonates or hydroxides either in aqueous solution or in powder form. The soluble alkali phosphate is removed by leaching. To obtain coloured pigments the titanium phosphate may be calcined with salts of a coloured base with a volatile acid, e.g., ferrous sulphate for red pigments or barium sulphate and ammonium dichromate for green pigments.

A. R. POWELL.

Manufacture of moulded products. L. H. HARTUNG and F. FOLBERT (U.S.P. 1,668,768, 8.5.28. Appl., 9.1.25).—Crude asbestos is mixed with water, zinc chloride, and soap, and the product moulded under pressure, dried, immersed in a hot soap solution, and again dried. Magnesite and diatomaceous earth may be added to the mix, also aluminium sulphate or sodium and potassium silicates to serve as binding and hardening agents. Resin as well as fatty soaps may be used, and treatment with aqueous calcium chloride renders the product more waterproof. After drying, it may be treated with alkali silicate to enable it to withstand high temperatures.

F. G. CLARKE.

Apparatus for the separation of hydrogen from hydrogenous [gas] mixtures [water-gas etc.]. H. M. R. BARJOT (B.P. 291,576, 14.4.27).—The gases are pumped through a combination of diffusion units consisting of discoid compartments having their flat faces constructed of porous material and surmounted by conical covers for receiving the gases diffusing through the faces. The units are so connected that the gases which have diffused through the porous partitions and those which have escaped diffusion flow separately into the central compartments of different units, the process being repeated until separation is complete. A modified form of apparatus comprises a single unit in the form of a double cone separated by porous partitions into a number of compartments each of which is connected with the adjacent compartments by external pipes provided with pumps, so that the gas passing upwards is progressively richer, and that passing downwards is progressively poorer in hydrogen.

L. A. COLES.

Production of absorbents for carbon dioxide, for use in respirators etc. C. A. F. KAHLBAUM

CHEM. FABR. G.M.B.H. (G.P. 447,425, 7.3.26).—Calcium oxide slaked with the theoretical quantity of water or previously prepared calcium hydroxide, alone or mixed with binding agents or fillers, *e.g.*, alkali hydroxides, salts, colloids, organic compounds, etc., is converted by heating or other means into a compact, easily granulated, hydrated product having a water content of 20–40%. L. A. COLES.

Red lead (B.P. 283,898).—See XIII.

VIII.—GLASS; CERAMICS.

Resistance of ordinary window-glass to the action of water. M. SCORY (Bull. Soc. chim. Belg., 1928, 37, 186–203).—The effect of water on glass is best determined from either the dissolved alkali or the total dissolved solids. Measurement of the loss of weight of the glass is unsatisfactory. As the process is simply the hydrolysis of silicates, an increase of silica should lessen the action; it should also vary with the heats of formation and solubilities of the different hydroxides. Slow cooling of the glass should favour resistance. In the manufacture of window glass by the Fourcault machine it is necessary to increase the soda content relative to lime (as compared with hand-working) in order to avoid devitrification; this decreases resistance to moisture. A better alternative is to use lime and magnesia in equimolecular proportions. The resistance of a large number of samples was determined by heating the powdered glass with water at 80° and determining the dissolved alkali by conductivity measurements. The degree of corrosion is approximately proportional to the square of the soda-lime ratio, treating magnesia as lime, and silica being constant. As a result, the desirable composition of window glass for the Fourcault machine has been determined as 72.5% SiO₂, 13.5% Na₂O, 10.5% CaO, 2.0% MgO, and 1.5% of impurities. This glass does not devitrify, works well, and is very resistant to atmospheric action. The silica content approaches closely to that indicated as desirable by Tscheuschner's formula. C. IRWIN.

Effects of carefully annealing optical glass. L. W. TILTON, A. N. FINN, and A. Q. TOOL (J. Amer. Ceram. Soc., 1928, 11, 292–295).—Deviations from the mean index of refraction in six well-annealed blanks from the same barium-flint melt were shown to be due to small differences in temperature during annealing. These variations in optical density were largely removed by preheating and re-annealing, provided that the temperature gradients in the annealing furnace were sufficiently low. F. SALT.

Elastic constants of glass. E. P. PERMAN and W. D. URRY (Proc. Physical Soc., 1928, 40, 186–192).—The compressibility coefficients of an alkali-lime-alumina-silicate glass and of Jena 16¹¹¹ glass, determined over the temperature range 30–80° by compressing a bulb containing water and closed by a graduated capillary, were $(238 + 0.185t) \times 10^8$ and $(220.6 + 0.28t) \times 10^8$, respectively. For the same glasses, by application of pressure only on a piezometric bulb, values for Poisson's ratio, Young's modulus, and rigidity modulus have also been determined. A. COUSEN.

Devitrification of old glass. H. R. CHIPMAN and D. MCINTOSH (Proc. Nova Scotian Inst. Sci., 1927, 16, 144–146).—Some Dumas bulbs 20–30 years old, and sealed at the time of manufacture, devitrified (even at 100°) on the outside only, showing that exposure to water vapour is essential for devitrification; also, by previously washing with hydrofluoric acid to remove silicic acid, devitrification was prevented. These observations support Germann's theory (A., 1921, ii, 262).

S. J. GREGG.

Characterisation of clay. A. F. JOSEPH (Trans. Ceram. Soc., 1928, 27, 1–11).—The term "clay" is restricted to that portion of the raw material of which the largest particles are 0.002 mm. The proportion of this true "clay" varied from 12% to 90% in twelve samples of ceramic clays. The properties of a clay depend on the nature rather than on the amount of the "clay" fraction present. The higher the silica: alumina ratio in the "clay" portion, the more highly developed are such properties as imbibitional power for water, heat of wetting, shrinkage, tensile strength, and absorptive power for vapours and dyes. The colloidal content of a raw clay is taken as identical with its "clay" content. F. SALT.

Drying of clay under definite conditions. I, II. III. Relation between drying rate and air condition. R. S. TROOP and F. WHEELER (Trans. Ceram. Soc., 1927, 26, 231–238, 239–248, 261–275).—I. A Stourbridge fireclay, after suitable treatment (wedging etc.), was moulded in cylindrical glass moulds, one end of each mould being sealed, so that drying of the clay took place from one end only. Six cylinders were treated at a time in a drying chamber and air previously conditioned was made to pass at a velocity of 0.5 m./min. over the exposed ends. Each experiment lasted 36 hrs., and a cylinder was removed every 6 hrs. and sampled along its length for moisture content. The relative humidity of the air was maintained constant at 90%, and the temperature was varied from 30° to 80° in steps of 10°.

II. Under the conditions of the experiments, evaporation of water followed the law: $W = A(P_s - P_a)$, where $A = 0.0158$. Tables and curves are given showing the distribution of water in the clay cylinders.

III. Experiments were carried out on Stourbridge fireclay, by the foregoing method, for air temperatures ranging from 30° to 80°, the humidity varying from 50% to 90%. The effect on the rate of drying of relative humidity, temperature, and velocity of the air was studied. With the clay used, the rate of drying during the first stage corresponded roughly with that to be expected from water under the same conditions; later the internal movement of the water within the clay controlled the rate of drying. F. SALT.

Factors influencing the thermal properties of minerals and products of the ceramic industry. W. M. COHN (J. Amer. Ceram. Soc., 1928, 11, 296–306).—A brief survey is presented of factors such as chemical composition, degree and duration of firing, rate of heating and cooling, porosity, grain size, etc., which affect the thermal properties of certain ceramic raw materials and fired bodies. An extensive bibliography is included. F. SALT.

Three fundamental principles of ceramics. H. H. STEPHENSON (Trans. Ceram. Soc., 1928, 27, 21—22).—The term thixotrope, used by Freundlich to indicate a colloid which changes in properties by mechanical treatment, is applied to clays. The behaviour of a ceramic body during and after heat-treatment is a function of the silica inversion. The chemistry of glazes is one of finished reactions; that of bodies one of arrested reactions. F. SALT.

Acid-resisting over-glaze colours. R. V. TAILBY (J. Amer. Ceram. Soc., 1928, 11, 307—309).—To meet modern requirements (mechanical washing etc.) on-glaze colours must have a coefficient of expansion closely approximate to that of the glaze, a maturing temperature not above cone 017, be capable of application in a uniform manner, particularly for transfer work, and be resistant to acids and alkalis. A suitable test for acid-resistance consists of immersing the ware in a 3% hydrochloric acid solution for 5 hrs. at 20°; 4% acetic acid and 5% caustic soda solution may also be used. F. SALT.

Methods for testing crazing of glazes caused by increases in size of ceramic bodies. H. G. SCHURECHT (J. Amer. Ceram. Soc., 1928, 11, 271—277).—The type of crazing which becomes apparent some considerable time after firing was investigated. Ware which had become crazed after 12 years' outdoor storage was found to have expanded, owing to hydration, by approximately 0.09%, whilst the glaze had remained constant in size. Methods of testing bodies for this type of crazing are: absorption after 2 hrs.' boiling and 24 hrs.' soaking in water; solubility in sulphuric acid; ignition loss above 110° after outdoor storage for 3 years; subjecting specimens to a steam pressure of 150—175 lb./in.² in an autoclave for 1 hr. Bodies having a low porosity, low solubility in sulphuric acid, and low ignition loss above 110° show the least tendency to produce this type of crazing. F. SALT.

Peeling of "slip-banded" ware. W. EMERY (Trans. Ceram. Soc., 1927, 26, 276—289).—A study was made of the fault known as "peeling" of the engobes applied in the form of bands and lines to coloured bodies (e.g., red teapots) or to earthenware bodies. Experiments were made on a plastic red-burning clay, a plastic buff-burning clay, and on an earthenware body; six different bases were used for the coloured slips. Mechanical adhesion, or bond, between the ware and the applied slip before firing, and the temperature-contraction range during firing are important factors. The greatest margin of safety was obtained with the red clay bodies. For the slips, mixtures having a comparatively low vitrifying temperature gave best results; a refractory slip (china clay) was least successful. For an earthenware body, the slip should contain about 10% of ball clay and 90% of dry earthenware body scraps. F. SALT.

Electrical pottery firing. B. J. MOORE and A. J. CAMPBELL (Trans. Ceram. Soc., 1928, 27, 12—20).—A "unit-size" electrical enamel tunnel kiln is described, which is about 82 ft. long and consists of an entrance zone, preheating zone, firing zone, and a cooling zone. The placing capacity of the trucks is 4 ft. 5 in. by

1 ft. 3 in. by 2 ft., and the propulsion is at the rate of one truck per hr. The method of B.P. 270,035 (B., 1927, 492) is used for installing the heating units in the kiln. Comparative costs of running and maintaining both electrical and coal-fired kilns are given.

F. SALT.

Influence of oxidising and reducing atmospheres on refractory materials. II. Experiments with a cone mixture containing added amounts of pure ferric oxide. A. E. J. VICKERS (Trans. Ceram. Soc., 1927, 26, 177—184; cf. B., 1925, 849).—The effect of heating such material in atmospheres of nitrogen, oxygen, air, carbon dioxide, carbon monoxide, sulphur dioxide, hydrogen, and water vapour was studied. Nitrogen, oxygen, and air have no detrimental effect; carbon dioxide is comparatively harmless. The other atmospheres produced a marked decrease in the refractoriness of the material, water vapour being particularly active in this respect. The effects produced appeared to be proportional to the amount of ferric oxide added. F. SALT.

Prevention of disintegration of blast-furnace linings. H. K. MITRA and A. SILVERMAN (J. Amer. Ceram. Soc., 1928, 11, 278—291).—A study was made of the disintegration of refractory materials due to "iron spots," the object being to identify, and prevent the action of, the substance in the "iron spots" which acts as catalyst. Spots of ferric oxide in a firebrick were found to be the original cause of disintegration of blast-furnace linings. Ferric oxide is reduced to ferrous oxide by carbon monoxide at 500°, and ferrous oxide is the catalyst which hastens the reaction $2\text{CO} \rightarrow \text{CO}_2 + \text{C}$, the deposited carbon being the cause of the disruption of the brick. Ferrosferric oxide is immune in this action; hence, by converting the ferric oxide in a brick into this oxide, disintegration is prevented. Such conversion is, however, extremely difficult. F. SALT.

Premature failure of combustion chamber material. A. J. DALE (Trans. Ceram. Soc., 1927, 26, 290—296).—The failure of a material containing 88% of silica in the combustion chamber of a gas-retort setting is examined and explained in the light of results of the ordinary refractory test and the refractoriness-under-load test carried out on the unused material. The latter test was made with loads of 50 lb., 10 lb., and 4 lb. per sq. in. It is concluded that combustion chamber material must have at least a moderate refractoriness, and there must be good cohesion between grog and matrix up to maximum working temperatures. The suitability of a material can be gauged from the results, properly interpreted, of two under-load tests (high and low loadings). F. SALT.

Use of the simulative test furnace as a means of making comparative tests of fire bricks. G. L. ROGERS (J. Amer. Ceram. Soc., 1928, 11, 323—329).—The U.S. Navy Simulative Service Test for refractories is conducted in small oil-fired furnaces. For purposes of comparison, one side wall of the combustion chamber is built up of bricks and cement of approved quality, and the other side wall of bricks and cement under test. A test consists of two runs of 24 hrs. each, at

furnace temperatures of 1590° and 1650°, respectively. After each run, a spalling test is conducted by injecting air at room temperature, under pressure, into the furnace immediately after the burner has been shut off.

F. SALT.

Open-hearth steel works' refractories. A. T. GREEN (Trans. Ceram. Soc., 1928, 27, 63—92).—The design and application of the open-hearth furnace are discussed. Silica bricks used in these furnaces must be sufficiently coarse in texture to counteract the tendency to spall, but the original grading should also contain a large proportion of "flour," which determines the matrix formation. Screen analyses of six bricks are given, and the suitability of such bricks for different parts of the furnace is discussed. Bricks having a sp. gr. of about 2.42 are most suitable; more highly converted material has a greater tendency to spall. The phenomena of corrosion and erosion, and the desirable properties and characteristics of refractories suitable for furnace doors, regenerators, chequer brickwork, and the ladle are discussed. Soaking pits and reheating furnaces are dealt with in a similar manner. Finally, the importance of careful storage of the refractories, under cover and properly enclosed, is pointed out. F. SALT.

Temperature diffusivity and thermal conductivity of silica refractory material at high temperatures. A. T. GREEN (Trans. Ceram. Soc., 1927, 26, 159—167, 168—176).—The temperature diffusivity and thermal conductivity were determined by the method previously described (B., 1927, 547) on nine different silica products used in industry. The relation between thermal conductivity on the one hand and texture, constitution, and degree of heat-treatment on the other was studied. The effect of a high firing temperature is to produce high coefficients of thermal conductivity and temperature diffusivity in modern silica products. A product containing a relatively high proportion of cristobalite had exceptionally good conductivity and high thermal diffusivity. High "permeable" porosity favoured a high rate of increase in diffusivity at high temperatures.

Comparative data are presented on the temperature diffusivity and thermal conductivity of silica and fire-clay (containing less than 75% SiO₂) materials. A measure of the rate of increase of diffusivity and conductivity is obtained by deducting the average values of the constants at 600°, 700°, . . . 1000° from the average values at 1000°, 1200°, and 1300°. In general, the diffusivities of silica products, at both lower and higher temperatures, are higher than those of fireclay, but individual samples of either material may vary considerably. The apparent sp. gr. of a well-fired silica brick is much less than that of a fireclay brick, and this tends to lower the coefficient of thermal conductivity of the silica product. Well-fired silica bricks are better conductors than firebricks at high temperatures (1200°); poorly burned silica bricks are comparatively poor conductors. At lower temperatures (800°) many silica bricks and firebricks have about the same conductivity. Silica bricks, in general, show a greater rate of increase of diffusivity and thermal conductivity with rise in temperature than firebricks. A material of low porosity is not necessarily a good conductor of heat; at high

temperatures the reverse is frequently true. The rate of increase of diffusivity and conductivity with temperature is very high in certain highly porous materials.

F. SALT.

Effects of temperature on the mechanical properties of silica products. A. J. DALE [with A. SCOTT] (Trans. Ceram. Soc., 1928, 27, 23—62).—Experiments were carried out on bricks made in the laboratory from different raw materials, on actual commercial products, and on selected clay-bonded silica bricks. Three different kinds of raw material were used in the laboratory bricks, and the effect of different gradings was studied. Not only the degree but also the homogeneity of conversion of the quartz is an important factor determining the temperature at which failure takes place under load. The effect of varying proportions of cristobalite, tridymite, and unconverted quartz in the fired bricks is noted. Lime-bonded bricks were more rigid at high temperatures than clay-bonded bricks. No clear relation was found between refractoriness, density, porosity, and chemical composition, and the behaviour of silica bricks under load at different temperatures; making, mixing, firing, and the nature of the bond, however, are important factors. It is suggested that two under-load tests (50 lb. and 4 lb. per sq. in.) would give a sufficiently clear indication of the thermal and mechanical properties of silica bricks.

F. SALT.

Sodium sulphate for glass. BESOMBE.—See VII.
Calcium-sodium ratio of water. HART.—See XXIII.

PATENT.

Electric [tunnel] kiln. T. C. and W. O. PROUTY (U.S.P. 1,671,742, 29.5.28. Appl., 27.6.27).—The kiln has heating elements mounted upon a conveyor and electrically operated during the passage of the ware through the kiln.

H. ROYAL-DAWSON.

IX.—BUILDING MATERIALS.

PATENTS.

Production of fused cements of predetermined setting time. STUDIENGES. P. NUTZBARMACHUNG DER SCHWEIZ. ERZLAGERSTÄTTEN (Swiss P. 119,656, 4.7.25).—The fused product is separated into portions which are cooled at different rates, the setting time decreasing as the time of cooling is increased. The portions are mixed before grinding in such proportions as to give a cement having the desired setting time. L. A. COLES.

Treatment of timber for the destruction of the borer, larvæ, beetles, or other pest. A. M. KOBILKE (U.S.P. 1,672,326, 5.6.28. Appl., 30.1.25. Austral., 14.2.24).—The timber is placed in a kiln, a vacuum created, and heat applied. By means of a vacuum pump gases are drawn from the timber into cooler zones on each side of the kiln, which are partially separated from the latter by condenser plates. When the timber is nearly dry the vacuum is released by the admission of poisonous fumes.

F. G. CLARKE.

Apparatus for mixing liquids and solids such as concrete or mortar. P. VOGLSAMER (B.P. 284,634, 1.2.28. Ger., 1.2.27).

Manufacture of asbestos-cement and like slabs or tubes. R. B. CHEDLER (B.P. 291,660, 30.9.27).

Veneering of brick. E. V. POSTON (B.P. 291,639, 2.8.27).

Cement for oil stills (B.P. 291,585).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Preparation and properties of silicon steel. C. WALLMANN (Stahl u. Eisen, 1928, 48, 817—821). H. KOPPENBERG (*Ibid.*, 821—822).—For the production of a high-silicon steel for constructional purposes the materials used must be relatively pure and almost free from phosphorus and sulphur, and, owing to the high viscosity of the steel, a high temperature and a long period in the furnace must be employed. This results in a larger fuel consumption and a shorter life for the refractories than is the case with ordinary steels. Silicon steels always solidify with a deep pipe, and therefore there is a large proportion of scrap from every ingot. Unless the heat-treatment during working is carried out slowly and carefully there is liability to loss by cracking either during the annealing or in the subsequent rolling operation. The tensile strength of hard-rolled sheets of silicon steel increases as the degree of rolling is increased from 54 kg./mm.² with 30 mm. thickness to 60 kg./mm.² with 9 mm. thickness. After annealing, the average tensile strength is 54—55 kg./mm.², and the yield point 36—38 kg./mm.² Koppenberg briefly reviews the difficulties met with in melting, casting, and working silicon steel, and covers similar ground to that of Wallmann.

A. R. POWELL.

Constitution of the iron-silicon alloys, particularly in connexion with the properties of corrosion-resisting alloys of this composition. M. G. CORSON (Amer. Inst. Min. Met. Eng. Tech. Pub., 1928, No. 96, 47 pp.).—A constitutional diagram, based on points of agreement in other published diagrams, is constructed. The formation of a compound Fe_3Si is reaffirmed. At 20% Si the alloys show a eutectic structure composed of Fe_3Si and FeSi . No evidence of the existence of Fe_2Si , Fe_3Si_2 , or FeSi_2 was found. The ductility, malleability, and resistance to corrosion of the alloys are discussed; the corrosion-resistance is attributed to the compound Fe_3Si .

CHEMICAL ABSTRACTS.

Rate of carbon elimination and degree of oxidation of the metal bath in basic open-hearth practice. A. L. FEILD (Amer. Inst. Min. Met. Eng. Tech. Pub., 1928, No. 111, 17 pp.).—Equations are given for the computation from operating data of the relation between rate of refining and degree of oxidation of the metal bath. Increased rate of refining appears to increase the concentration of ferrous oxide in the metal. If two of the three factors, carbon content, ferrous oxide content of slag, and temperature, are known, the third may, under certain conditions, be determined from tabulated data.

CHEMICAL ABSTRACTS.

Production of high-alumina slags in the blast furnace. T. L. JOSEPH, S. P. KINNEY, and C. E. WOOD (Amer. Inst. Min. Met. Eng. Tech. Pub., 1928, No. 112, 20 pp.).—In general, slags containing nearly equal

amounts of alumina and lime have the lowest m.p. when the percentage of calcium is higher than that of alumina. Calcium aluminates desulphurise metal rapidly at 1500—1520°.

CHEMICAL ABSTRACTS.

Hardening of carbon steels by deformation; effect of temperature, time, and structure. F. SAUERWALD, L. MICHALSKY, R. KRAICZEK, and G. NEUENDORFF (Arch. Eisenhüttenw., 1927—8, 1, 717—720; Stahl u. Eisen, 1928, 48, 770—771).—"Cold" deformation of metals causes hardening and the formation of a fibrous structure; "hot" deformation, in the strict sense, produces no hardening effects, but causes the metal to recrystallise spontaneously. For pearlitic and hyper-eutectoidal steels the region of "cold" deformation extends to the pearlite line in the iron-carbon diagram, whereas the γ -solid solution region is entirely a region of "hot" deformation in which rapid deformation, e.g., hammering, produces immediate and complete recrystallisation. The temperature range of "blue brittleness" is also the range in which cold deformation produces the maximum hardening effect.

A. R. POWELL.

Electrochemical action of inhibitors in the acid dissolution of steel and iron. E. L. CHAPPELL, B. E. ROETHEL, and B. Y. MCCARTHY (Ind. Eng. Chem., 1928, 20, 582—587).—Results of fundamental experiments on the pickling of iron and steel, using quinoline ethiodide as a standard inhibitor, showed that the rate of hydrogen evolution decreased with increasing concentration of inhibitor, which also caused a regular decrease in the anode potential. This decrease is, however, believed to be too small to account for the lower rate of evolution of hydrogen. The cathode potential increases with increasing current density and, on the addition of inhibitors, with a reverse effect at higher temperatures. Increase of overvoltage varies with the metal and the condition of the surface; e.g., Armco iron, steel, and white cast iron are more resistant to acid attack than malleable and wrought iron on the surfaces of which inhibitors give a less overvoltage increase. When etching polished steel the gas was evolved principally from the grain boundaries and scratch marks, and, in iron, from slag areas also. It is considered that when immersed in acid iron enters into solution at the anode areas and discharges hydrogen ions at the cathode areas defined by the narrow spaces of the grain boundaries and slag areas. Most inhibitors being either bases or positively charged colloids travel to the cathode with the hydrogen and form a protective layer upon the surface of the iron.

C. A. KING.

Determination of aluminium in steel. W. P. KUEHLER and S. SHANEMAN (Chemist-Analyst, 1928, 17, 4).—The hydrochloric acid solution, free from silica, is boiled with sodium phosphate, the hot solution neutralised with ammonia, boiled with sulphurous (and, if necessary, hydrochloric) acid, and precipitated with acetic acid and ammonium acetate. The precipitate is dissolved in hydrochloric acid, oxidised with nitric acid, and the iron precipitated with sodium hydroxide. The aluminium in the filtrate is then precipitated with ammonia and hydrogen peroxide, or (after evaporation with nitric acid and oxidation of chromium with

potassium chlorate) with ammonia and ammonium carbonate.

CHEMICAL ABSTRACTS.

Application of a high-vacuum induction furnace to the study of gases in metals. P. H. BRACE and N. A. ZIEGLER (Amer. Inst. Min. Met. Eng. Tech. Pub., 1928, No. 59, 21 pp.).—Iron, contained in zircon or alumina crucibles in a high-frequency induction furnace in a vacuum, was melted and the gases were analysed, allowance (10%) being made for gases evolved from the apparatus. Electrolytic iron evolved 25% more gas than Armco iron, including water, but no nitrogen from the former. Carbon and oxygen were present in constant ratio to one another and to the total gas. A marked evolution of gas shortly before melting was probably due to greater solubility of the gases in γ - than in δ -iron.

CHEMICAL ABSTRACTS.

Influence of certain elements present in steel on the determination of oxygen in steel by the hydrogen reduction method. P. BARDENHEUER and C. A. MÜLLER (Arch. Eisenhüttenw., 1927-8, 1, 707-712; Stahl u. Eisen, 1928, 48, 795).—Determination of oxygen in steel by heating the turnings in hydrogen and collecting the water formed in phosphorus pentoxide leads to high results if the steel contains nitrogen unless the sample is mixed with an equal weight of pure antimony before treatment. A high content of phosphorus leads to low results, but sulphur is without influence. The oxygen content due to included silica, alumina, and other oxides not reduced by hydrogen cannot be determined by this method, but must be determined separately by the bromine or iodine procedure (B., 1927, 966) or by the method of Hessenbruch and Oberhoffer (B., 1928, 409).

A. R. POWELL.

Determination of traces of antimony in copper. H. BLUMENTHAL (Z. anal. Chem., 1928, 74, 33-39; cf. A., 1922, ii, 231).—A solution of the metal in nitric acid (*d* 1.4) is diluted and dilute ammonia added as long as any precipitate continues to be formed. The latter is dissolved in a little nitric acid, 5 c.c. of a 5% manganous sulphate solution are added, followed by 3 c.c. of *N*-potassium permanganate, and the mixture is heated with agitation. The liquor becomes decolorised and the manganese dioxide precipitated in a state of conglomeration. A further 3 c.c. of permanganate solution are added, agitation and heating being continued, followed by filtration and a rapid wash with hot water. The clear filtrate is once more similarly treated, and the precipitates of manganese dioxide are combined and washed down with dilute hydrochloric acid containing a little hydrogen peroxide before being brought completely into solution by heating with hydrochloric acid till chlorine is no longer evolved. The insoluble residue is collected on a filter, washed, mixed with sodium peroxide, and fused, the melt being taken up in water. After acidifying with hydrochloric acid it is combined with the last filtrate. After neutralisation of free acid with ammonia, hydrogen sulphide is used to precipitate all the antimony together with tin, arsenic, bismuth, lead, and some copper. This precipitate is collected, washed, extracted with hot sodium sulphide, the solution filtered, and the filtrate containing the sulphides of antimony, tin, and arsenic reprecipitated with dilute sulphuric acid, filtered, and

dissolved in hot brominated hydrochloric acid. The separation and determination of the antimony are then carried out by the usual methods. R. A. PRATT.

British standard specification for chemical lead (Brit. Engineering Standards Assoc., 1928, No. 334, 18 pp.).—The standard lead shall contain 99.99% Pb (up to 0.05% of which may be replaced by copper), and the content of impurities shall not exceed the following: 0.002% Ag, 0.005% Bi, 0.003% Fe, 0.002% Sb, 0.002% Zn, 0.001% (Ni + Co), and only traces of tin, cadmium, and arsenic. The flash and aqua regia tests for lead are described. These are recommended as useful indicators subsidiary to complete analysis, a detailed scheme for which is set out with the comparative results obtained by five analysts for one sample. Other analyses of different leads are given together with flash points (ranging from 315° to 225°) and results of the aqua regia test. Whilst in general lower flash point and poor results with aqua regia are associated with excessive impurities (bismuth and antimony), in some cases the results are quite anomalous.

C. IRWIN.

British standard specification for regulus metal (Brit. Engineering Standards Assoc., 1928, No. 335, 9 pp.).—The lead used shall not contain more than 0.1% of impurities other than antimony, nor the antimony more than 0.5% of impurities other than lead. Four types of regulus metal are specified, viz., 6-8% regulus for tanks and containing vessels, 8-10% metal for valves, plugs, and working parts of pumps etc., 10-12% metal for screws or other parts requiring machining, and metal containing over 12% of antimony. For all these alloys the contents of arsenic and zinc should not exceed 0.01% of each, copper and tin 0.10% of each, and sulphur 0.02%. The minimum ultimate tensile strength of the four alloys and their Brinell hardness numbers are specified. Bend tests are also described which are, however, chiefly of value for 6-8% regulus. The recommended analytical methods are subjoined.

C. IRWIN.

Electrolytic refining of copper, using complex salt of cuprous chloride. I, II. N. KAMEYAMA and T. NODA (J. Soc. Chem. Ind. Japan, 1927, 30, 773; 1928, 31, 343).—I. As an electrolyte the use of a cuprous salt instead of the usual cupric sulphate for the purpose of saving electrical power consumption was tried. An aqueous solution of potassium chloride nearly saturated with cuprous chloride was found advantageous at 50-75°, such a solution containing about 4% Cu; a little gelatin and about 2% of hydrochloric acid should be added. Nitrogen was passed into the electrolytic bath in order to stir it and exclude air therefrom. A current density of about 3 amp./dm.² was possible with high current efficiency and without giving a bad cathodic deposit; the anode used was pure copper. When impure copper (98.8% Cu) was used a cathode copper of 99.92% purity was obtained.

II. Aqueous solutions of sodium chloride and calcium chloride were used for the dissolution of cuprous chloride, and the solutions obtained were used as electrolytes for the refining of copper. These electrolytes were found just as suitable as those in which potassium

chloride was used, and even preferable when higher concentrations of copper in the electrolyte are required.

S. OKA.

Hydrogen in electrolytic zinc. W. R. ELLIOT (Proc. Nova Scotian Inst. Sci., 1927, 16, 152—154).—The zinc was placed in a pyrex tube connected to a Toepler pump, exhausted to 0.01 mm., and allowed to remain for some hours; it was then heated to 200° and finally melted, and a total of 0.5—1 c.c. of hydrogen per g. of zinc was evolved (according to the sample). The following stages were observed in one typical case: during exhaustion, 13% of gas; after 24 hrs., 9%; on heating to 200°, 11%; and on melting, 67%. Both laboratory and commercial samples gave similar results. These figures are tenfold smaller than those of Schwarz (cf. Ralston, B., 1925, 361), consequently the pressures developed inside the zinc are small.

S. J. GREGG.

Reduction roasting, leaching, and electrolytic treatment of Bolivian tin concentrates. C. G. FRNK and C. L. MANTELL (Eng. Min. J., 1928, 125, 452—455).—Gaseous reduction of the concentrate is followed by leaching with a solution containing sulphuric acid (15%) and sodium chloride (5%); tin is recovered (95—98%) electrolytically.

CHEMICAL ABSTRACTS.

Blast-furnace linings. MITRA and SILVERMAN. **Steel works' refractories.** GREEN.—See VIII.

PATENTS.

[Multiple-hearth] furnace construction and operation. D. BAIRD, Assr. to NICHOLS COPPER CO. (U.S.P. 1,671,395, 29.5.28. Appl., 12.3.26).—In a mechanically-rabblled, multiple-hearth, roasting furnace the individual hearths have discharge openings alternately at the centre and at the periphery, and at least one of the hearths having an opening at the periphery has also an opening at the centre to provide a direct gas passage to the other hearths partially independent of the path of the material being treated.

A. R. POWELL.

Shaft furnaces [cupolas]. G. SIMON (B.P. 291,258, 21.7.27).—A vertical shaft is connected at its lower end by means of a channel to a fore-hearth above which is the recuperator, the connexion between the two being provided with adjustable dampers. The fore-hearth is provided with a slag overflow at the top, and at the bottom with an upwardly inclined conduit communicating with an adjacent collecting chamber for the metal. This chamber is heated by means of liquid fuel, the flames and products of combustion from which pass over the fore-hearth and upwards through the vertical shaft thereby assisting the melting operation therein. [Stat. ref.]

A. R. POWELL.

Semi-muffle forge furnace. W. McM. HEPBURN, Assr. to SURFACE COMBUSTION CO. (U.S.P. 1,672,863, 5.6.28. Appl., 30.4.27).—The goods are heated first by direct contact with gases freely circulating in a heating zone, and then for final heating pass into a passage open to the heating gases but from which the heating gases are excluded by another fluid. B. M. VENABLES.

Muffle furnaces for annealing. GIBBONS BROS., LTD., M. VAN MARLE, and T. E. BRIDGFORD (B.P.

291,580, 21.4.27).—The ingress of air to a muffle annealing furnace is prevented by providing a continuous cascade of curtain of oil or water at the inlet and outlet of the furnace, in such a way that the articles to be annealed have to pass through the liquid curtains on entering and leaving the furnace. The curtains are produced by allowing liquid to fall continuously from an upper tank against a stream of liquid projected upwardly under pressure from a lower tank, the liquid being kept in circulation by means of suitable pumps.

A. R. POWELL.

Charging of high-frequency melting furnaces. HIRSCH, KUPFER- & MESSING-WERKE A.-G. (B.P. 282,656, 12.12.27. Ger., 23.12.26).—Metal to be treated is packed, outside the furnace, in a metal basket, preferably composed of the same metal as the charge or containing one or more constituents of the charge. The basket melts with the charge.

J. S. G. THOMAS.

Concentrating tables. A. R. HARVEY (B.P. 291,498, 26.2.27).—The table is hung by link work and is provided with a number of riffle trays. It is shaken by a rotating crank which has lost motion either in the crank bearing of or on the small end of the connecting rod. Side sway may also be introduced by shaping the ends of the crank bearing as cams.

B. M. VENABLES.

Roasting of [tin] ores and oxidation of pyritic and sulphide ores etc. F. L. WILDER, E. MORRIS, E. SCHIFF, and E. S. KING (B.P. 291,568, 6.4.27).—The ore is mixed with an aluminous binding material, e.g., china clay, and ferrous sulphate solution, and the mixture is formed into ring-shaped briquettes for roasting in vertical retorts or kilns. When the ore contains a deficiency of sulphur, iron pyrites is added to the ore-clay mixture. Tin ores or residues are ground to pass 200-mesh and made into a plastic mass with about one fifth their volume of china clay and sufficient of a 10% ferrous sulphate solution to provide 2.5% of ferrous sulphate in the dried product. The mass is briquetted, roasted, and subsequently treated to recover a high-grade tin concentrate by leaching, oil flotation, or the like.

A. R. POWELL.

Manufacture of steel in the Siemens-Martin furnace. F. SIEMENS A.-G., R. DURRER, F. C. SIEMENS, and A. SPRENGER (B.P. 291,717, 24.12.26).—The furnace is fired with blast-furnace gas into which a stream of poor coal, coke, semi-coke, or dust from a coal poor in gas is introduced, and the air is preheated by means of the waste heat from the furnace. During the fining period a stream of fine coal dust is blown with air over the surface of the molten metal below the stream of heating gases so as to form a protective layer, and after the fining period a stream of coal dust without air is fed on to the charge; impurities are then allowed to separate.

A. R. POWELL.

[Cast iron] ingot mould. E. R. WILLIAMS, Assr. to VULCAN MOLD & IRON CO. (U.S.P. 1,672,479, 5.6.28. Appl., 20.6.27).—A cast-iron mould having a structure composed of ferrite, graphite, and other constituents, such that the content of combined carbon is 0.7—1.1% has a low permanent expansion after repeated heating and cooling.

A. R. POWELL.

Production of rimmed steel ingots. A. T. CAPE, Assr. to MIDWEST METALLURGICAL CORP. (U.S.P. 1,672,446, 5.6.28. Appl., 8.2.27).—The structure and surface appearance of rimming steel are improved by adding 1–6 oz. of a fluoride to 3000 lb. of molten steel immediately after tapping it from the furnace.

A. R. POWELL.

Heat-treatment of metal [iron or steel] articles. A. HILGER, LTD., and F. TWYMAN (B.P. 291,483, 27.1.27).

—The article is made the whole or part of the secondary winding of a transformer, to the primary of which is applied alternating current of low frequency and, when the article is heated to the requisite temperature, a quenching medium is applied to the part to be hardened while the rate of cooling of the other parts of the article is retarded by maintaining part or the whole of the electrical heating.

A. R. POWELL.

Annealing of articles of iron or steel. Apparatus for use in annealing metal articles. V. DEMMER (B.P. 268,740 and 291,718, 3.3.27. Ger., [A] 31.3.26).—

(A) The articles to be annealed are placed in a sheet-iron box, the top is welded on except for a small vent, the box is exhausted of air, and the vent sealed. Alternatively, the box is heated to the maximum annealing temperature and the vent sealed with or without replacing the air by an inert gas. (B) A container suitable for use in the above bright-annealing process is described.

A. R. POWELL.

Coating of iron articles with a good heat-conducting metal. F. KUNZ (Swiss P. 118,977, 11.11.25. Ger., 17.7.25).—The iron article is dipped into a molten bath of the fluorides of sodium, potassium, calcium, barium, and manganese until the carbon is removed from the surface layers, then into a molten alloy of aluminium with zinc, magnesium, and manganese, whereby a coating of the alloy adheres tightly to the iron surface owing to the rapidity with which it diffuses into the ferrite layer produced in the fluoride bath.

A. R. POWELL.

Production of alloys for the construction or lining of apparatus etc. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 291,522, 3.3.27).—Pure alloys of iron and nickel free from sulphur and phosphorus are prepared by melting with the desired alloying elements the metal powder obtained by decomposition of the corresponding carbonyl. The operation is preferably carried out in a high-frequency induction furnace.

A. R. POWELL.

Tinning of [metal] containers. W. RAUCH and H. SANDREUTER (Swiss P. 119,996, 28.12.25).—The articles are pickled in hydrochloric acid (containing copper sulphate if the articles are made of a ferrous metal), coated with a paste of ammonium chloride, hydrochloric acid, and zinc and tin powders, heated until the paste melts, and finally washed to remove excess.

A. R. POWELL.

Manufacture of protective covering for aluminium bodies. W. C. PETERSON, Assr. to PACKARD MOTOR CAR Co. (U.S.P. 1,672,280, 5.6.28. Appl., 8.5.20).—Phenol and formaldehyde are heated until the volume of the mixture is reduced to one half, and it attains the consistency of molasses; an aluminium

silicate is then added in the proportion of 10% by wt. of the final mixture.

F. G. CLARKE.

Production of corrosion-resistant surfaces on metals. F. M. BECKET, Assr. to ELECTRO-METALLURGICAL Co. (U.S.P. 1,672,444, 5.6.28. Appl., 17.3.26).—The surfaces of iron and steel articles are rendered highly resistant to corrosion by heating them in contact with a finely-divided iron alloy containing chromium and silicon until cementation takes place.

A. R. POWELL.

[Alloy for] bells. K. MUCHENBERGER, SEN. (Swiss P. 118,348, 24.3.26).—The alloy contains 92% Al, 6% Cu, 1.5% Sn, and 0.5% Ag.

A. R. POWELL.

Decomposition of chromium ores with acids. P. ASKENASY and W. MOHRSCULZ (G.P. 447,537, 29.12.25).—The ore (chromite) is treated with an alkaline solution or with a basic substance prior to the acid treatment for dissolving the chromium but after any necessary acid treatment for the removal of soluble impurities.

A. R. POWELL.

Purification of metal [uranium or thorium] powder. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP Co. (U.S.P. 1,665,635, 10.4.28. Appl., 21.8.22).—The powder obtained by reducing the chloride of the metal with metallic sodium or calcium is washed with a solution of hydrogen chloride in methyl alcohol.

A. R. POWELL.

Alloys of high m.p. A. KROFF (B.P. 264,528 and 270,640, 15.1.27. Ger., [A] 16.1.26, [B] 10.5.26).—(A) Hard, tenacious alloys suitable for cutting tools are made by melting in a carbon resistance furnace a mixture of 5–80% of tungsten, 90–20% of molybdenum, and 5–0.5% of carbon. Part or all of the tungsten and/or part of the molybdenum may be replaced by tantalum, and 0.5–20% of cobalt, uranium, chromium, vanadium, titanium, zirconium, or silicon either alone or together may also be added. (B) Alloys containing 5–80% Ta, 5–0.8% C, and the remainder tungsten are claimed. Part of the constituents may be replaced by up to 25% of one of the metals mentioned in (A), and the tantalum may be replaced by a tantalum-chromium alloy containing up to 40% Cr.

A. R. POWELL.

Desilverising of lead bullion. B. M. O'HARRA, Assr. to AMER. SMELTING & REFINING Co. (U.S.P. 1,672,465, 5.6.28. Appl., 9.2.27).—Argentiferous lead is melted with zinc under a layer of a halide salt which dissolves the metal oxides present to form a slag, and the mixture is allowed to cool until the upper layer of zinc-silver alloy has solidified. The lower liquid layer of lead is removed and both layers are refined by known methods.

A. R. POWELL.

Preparation of articles for nickel plating. L. F. NELISSEN (B.P. 282,748, 17.12.27. Belg., 30.12.26).—A sufficient thickness of copper is deposited upon an oxidisable metal, e.g., steel, to ensure that pores etc. in the metal are filled with copper forced into them during the subsequent polishing operation.

J. S. G. THOMAS.

Nickel-plating of aluminium articles. R. MÜRSET (Swiss P. 118,976, 12.12.25).—The articles are pickled in a boiling solution of sodium carbonate, brushed with

a dilute solution of citric acid, potassium hydrogen tartrate, or aluminium acetate, washed in hot water, and plated in a nickel citrate or borate bath.

A. R. POWELL.

Chromium-plating of iron articles. STUDIENGES. F. WIRTS. U. IND. M.B.H. (Swiss P. 120,265, 18.9.25. Ger., 11.8.25).—The electrolyte comprises a solution containing chromic sulphate and chromium silicofluoride or borofluoride, and lead or graphite anodes are used. The bath is operated at 40–50°.

A. R. POWELL.

Providing a resistant coating upon surfaces of aluminium or its alloys. C. H. R. GOWER and S. O'BRIEN & PARTNERS, LTD. (B.P. 290,901, 20.10.27).—The article is subjected to anodic treatment at 15–20° in a bath containing sulphuric acid, together with potassium dichromate, sodium thiosulphate, lead acetate, barium sulphite, or ammonium or potassium thiocyanate. A darker coating is obtained by the addition of an ammonium salt to the bath, and a thicker coating by repeated treatment in the same or a different bath with intermediate periods of exposure to the air.

A. R. POWELL.

Electroplating the surface of aluminium or its alloys. C. H. R. GOWER and S. O'BRIEN & PARTNERS, LTD. (B.P. 290,903, 25.10.27).—The article is first made an anode in a solution containing sulphuric and hydrochloric acids or a sulphate or chloride, then washed and treated anodically in a solution containing potassium hydroxide and ammonia. Finally it is plated in any of the usual baths with copper, nickel, chromium, etc.

A. R. POWELL.

Cold-rolling of bands and strips from metals and alloys. O. BUSSE (B.P. 291,323, 21.1.28. Ger., 3.12.27).

Washing of ores (U.S.P. 1,672,448).—See I. **Paint for iron or steel (B.P. 291,486).**—See XIII.

XI.—ELECTROTECHNICS.

Valve-maintained high-frequency induction furnace; performance of induction furnaces. G. E. BELL (Proc. Physical Soc., 1928, 40, 193–203).—The furnace, in the design of which a modification of the Hartley circuit, employing a centre-tapped coil for the valve oscillator, was used, melted kg. charges of palladium, chromium, nickel, and iron with about 8.5 kw. input. In a mathematical analysis of the problem of heating by high-frequency induction, the energy values deduced agreed well with those obtained experimentally with the furnace.

A. COUSEN.

Breakdown of solid insulators. W. WERNER (Ann. Physik, 1928, [iv], 86, 95–128).—A method is described for investigating the breakdown potential of various insulators. The substances used were mica, glass, rock salt, sulphur, celluloid, etc. The breakdown potential was determined for several thicknesses of each substance, except in the case of celluloid, when only one thickness was used. The experiments were carried out in oil at room temperature, and in liquid air and liquid hydrogen. It was found that, in general, the breakdown potential was considerably less in liquid air than in oil. Exceptions, however, are noted. The

results when plotted on a graph between breakdown voltage and thickness give, for experiments in oil and in liquid air, straight lines of which the equation is $U = ad + b$, where U is the breakdown voltage, d is the thickness of the dielectric, and a and b are constants. The discharge takes place first through the medium. Breakdown never occurs between the points of contact of the electrodes with the dielectric, although the field strength is greatest here. It is due not to the presence of ions in the insulator, but to the fact that all substances contain gases. There is a good deal of evidence to show that the discharge is a gas-discharge. At low temperatures all substances become brittle, and it is considered that this brittleness has some connexion with the lower breakdown voltage at the temperature of liquid air. The photographs of mica pierced by the discharge show the same lines as are obtained when a sheet of mica is gently punctured by means of a needle. Experiments were also carried out with dielectrics in a high vacuum. Previous results on the breakdown voltage of insulators are too low for dielectrics free from gases. A. J. MEE.

Photogalvanic cell furnished with silver iodide electrodes, and its application to photometry and illuminometry. S. IIMORI and T. TAKEBE (Sci. Papers Inst. Phys. Chem. Res. Tokyo, 1928, 8, 131–160).—The photoelectric phenomena associated with the illumination of one electrode of a photogalvanic cell containing electrodes of silver iodide (the so-called Becquerel effect) are investigated, and attributed principally to the construction of the electrode film. The practical application of the cell in photometry is briefly discussed.

J. S. G. THOMAS.

Preparation of chromium plating solution. Physicochemical studies of the solution. Phenomena taking place during electrolysis. Y. KATO, T. MURAKAMI, and S. SAITO (J. Soc. Chem. Ind. Japan, 1928, 31, 128–131).—The solution is generally prepared from chromic acid solution by partial reduction or by addition of chromic hydroxide. It is recommended that chromic acid solution should be treated with insoluble organic colloids (cf. Jap. P. 71,950). In plating, a film of chromic chromate is first formed on the surface of the cathode, and during electrolysis chromic acid is reduced to chromic hydroxide, a film of which forms when the concentration of the latter becomes sufficiently high near the cathode. From the results of ultra-filtration and diffusion experiments it is confirmed that the chromic chromate solution is not colloidal. F.p. depression and electric conductivity measurements of the solution show that chromic chromate resembles the sulphate when dissolved in water. The electrical condition through the plating solution is therefore concluded to be one of electrolysis and not of cataphoresis, as is sometimes supposed. K. KASHIMA.

Precipitation of tar. SEIDENSCHNUR and GROH.—See II. **Fixation of nitrogen.** BRINER.—See VII. **Pottery firing.** MOORE and CAMPBELL.—See VIII. **Electrolytic copper.** KAMEYAMA and NODA. **Bolivian tin concentrates.** FINK and MANTELL.—See X.

PATENTS.

Electric furnace. H. L. WATSON, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,672,667, 5.6.28. Appl., 13.11.26).

—A continuous resistor comprising refractory bars arranged vertically in spaced relation, joined at their upper and lower ends by cross-pieces and resting on the lower cross-pieces, extends along the side wall inside the furnace chamber.
J. S. G. THOMAS.

Electric vacuum furnaces. [A] M. D. SARBEY, [B] G. D. BAGLEY, Assrs. to KEMET LABORATORIES CO., INC. (U.S.P. 1,671,451 and 1,671,461, 29.5.28. Appl., [A] 1.12.25, [B] 28.4.27).—(A) An accessible device for supporting a charge is arranged within a furnace chamber, adapted for operations under high vacua, sealed by a barometric column, and provided with a conduit. (B) A dense, non-porous screen surrounds a resistor within a gas-tight chamber.
J. S. G. THOMAS.

Manufacture of electric resistances. S. G. S. DICKER. From N. V. PHILIPS' GLOELAMPENFABR. (B.P. 291,591, 5.5.27).—A wire, preferably of a highly refractory metal, is disintegrated within an exhausted tube of insulating material through which leading-in wires, preferably of ferrochrome, are led at definite distances apart, and then definite lengths are cut from the tube. The exhausted tube may, if desired, be filled with a neutral gas.
J. S. G. THOMAS.

Non-aqueous electrolytic condenser. E. W. ENGLE, Assr. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,672,714, 5.6.28. Appl., 29.8.27).—An electrolytic cell contains an electrolyte consisting of a solution of a dissociable salt in an anhydrous alcohol, e.g., glycerol, below a layer of oil, and two electrodes, one of which is an aluminium plate provided with a protective film.
A. R. POWELL.

Manufacture of [wooden] separators [for accumulators]. W. S. GOULD and W. B. OSBORNE, Assrs. to GOULD STORAGE BATTERY CO., INC. (U.S.P. 1,666,007, 10.4.28. Appl., 23.10.23).—Flat strips of wood are heated for 8 hrs. at 100° in 3% sodium hydroxide solution, washed, immersed in sulphuric acid (*d* 1.02) until neutral, and dried.
A. R. POWELL.

Electric resistance. G. EGLY, Assr. to GEBR. SIEMENS & Co. (U.S.P. 1,671,469, 29.5.28. Appl., 31.3.25. Ger., 12.5.24).—See Swiss P. 116,016; B., 1927, 257.

Cathodes for mercury vapour apparatus [rectifiers]. W. DÄLLENBACH (B.P. 291,683, 24.11.27).

Insulating composition (B.P. 290,886).—See II. **Heat-treatment of metals** (B.P. 291,483). **Nickel plating** (B.P. 282,748 and Swiss P. 118,976). **Chromium plating** (Swiss P. 120,265). **Plating on aluminium or its alloys** (B.P. 290,901 and 290,903).—See X.

XII.—FATS; OILS; WAXES.

Combined method of determining soluble and insoluble volatile fatty acids in butter and fats. E. TCHÉTEROV (Bull. Soc. chim. Belg., 1928, 37, 153—163).—In the method described the saponification, Reichert–Meissl, butyric acid, and Polenske values can be determined successively on one 5 g. portion of the sample. After determining the saponif. value, using alcoholic potash, the higher fatty acids are removed as insoluble magnesium soaps and the alcohol is distilled

off from half (75 c.c.) of the filtrate. The Reichert–Meissl and Polenske values are determined on this portion. For the butyric acid value a slight modification of van Raalte's xylene method (cf. B., 1926, 563) is used, the determination being made on the solution used for the Reichert–Meissl number. The process was tested on various samples of butter and synthetic mixtures of butter with other fats with satisfactory results. From the data obtained, the average xylene value for butter by the method described is taken as 22.
W. J. BOYD.

Detection of decomposition in fatty oils and other fats. J. STAMM (Pharmacia, 1926, [5]; Chem. Zentr., 1927, II, 1520—1521).—Ten drops of the fatty oil are mixed with 5 drops of a suspension of 0.1 g. of finely-powdered *s*-diphenylcarbazine in 10 g. of vaseline oil, and the mixture is warmed for 3 min. in a boiling-water bath and then cooled. A control is carried out with vaseline oil alone and solid fats are dissolved in an equal quantity of vaseline oil. The degree of rancidity in the sample is judged from the intensity of the red colour produced in the reaction together with the odour and taste of the oil.
E. H. SHARPLES.

Working-up of soap lyes. J. GROSSER (Allgem. Öl- u. Fett-Ztg., 1927, 24, 375—376, 387—388, 399—400; Chem. Zentr., 1927, II, 1520).—Mechanical impurities are removed by filtration, hydrochloric or sulphuric acid is then added until the liquid is only faintly alkaline, and the mixture is boiled to remove carbon dioxide. Resinous and tarry substances are skimmed off, aluminium sulphate is added, and the liquid is boiled again. After removal of the precipitated solids the liquid is distilled in a vacuum evaporator.
E. H. SHARPLES.

Determination of iodine value [of fats]. E. LINDENBERG (Bol. Soc. Chim. São Paulo, 1928, 1, 92).—The iodine value of sparingly soluble or solid fats may be determined conveniently by Margosches' method (B., 1924, 639) by using alcohol containing 1—2% of hydrochloric acid, by boiling the fat with alcohol, and then cooling to 25°, or by adding 5—10% of chloroform to the alcohol used.
R. K. CALLOW.

Differentiation of [vegetable] oils by P. Jäger's "flow method." H. VON NABELL (Chem. Umschau, 1928, 35, 121—123).—The method of differentiation of oils due to Jäger ("Fließverfahren zur Unterscheidung pflanzlicher Öele," Stuttgart, 1927) depends on the characteristic phenomena observed when oils are dropped on suitable cardboard of specified nature, which is subsequently immersed in cold water for a brief period. The results obtained in this way with linseed oil, boiled oil, and tung oil (dealt with in Jäger's original publication) are reproduced and discussed, and a further series of photographs illustrate the characteristics of other oils and mixtures.
S. S. WOOLF.

Differentiation of fatty oils by the [Jäger] "flow method." J. SCHEIBER (Chem. Umschau, 1928, 35, 123—125).—Jäger's "flow method" (cf. preceding abstract) is adversely criticised. It is suggested that the "stains" produced be examined by transmitted light. The phenomena observed can be divided into three types, into which, however, the different classes of oil do not

regularly fall. It is pointed out that whilst tung oil and linseed oil show distinct characteristics in this test, the stain produced by tung oil that has been treated so that it does not exhibit the normal frosting effect (*e.g.*, by addition of β -naphthol) is indistinguishable from the linseed oil stain. S. S. WOOLF.

Detection of the decomposition of linseed oil by the diphenylcarbazine reaction of Stamm and the Fellenberg test. E. JÄGERHORN (Pharmacia, 1926, [5]; Chem. Zentr., 1927, II, 1521).—Linseed oil, acid value 3.6, after storage under different conditions, has been examined by Stamm's diphenylcarbazine reaction and by Fellenberg's fuchsin reaction. Oil exposed to light in open vessels after 80 days gave a weakly positive reaction in Stamm's test, after 180 days a rancid taste and smell with a still faintly positive reaction, and after 245 days' exposure decomposition was strongly advanced. Samples kept in the dark, in either open or closed vessels, gave negative Stamm reactions, and filtered and unfiltered oils decomposed at the same rate as a sample which had been heated at 100° for 1 hr. before storage. A positive reaction in the Fellenberg test was observed in open samples kept in the dark and samples exposed to the light, after 42 days. Linseed oil for pharmaceutical purposes should give no diphenylcarbazine reaction. E. H. SHARPLES.

Composition of normal oleines. M. DITTMER (Chem. Umschau, 1928, 35, 150—152).—Discrepancies are noted between the results of analysis of oleines by the usual methods, checked by a lead salt-alcohol separation, and the composition as deduced from the Kaufmann thiocyanogen value, the latter giving too high a proportion of linoleic and saturated acids. The author recommends that, while these contradictions remain unexplained, the Mackey value and resinification test should be used as criteria of the oxidisability of oleines (*cf.* Seifensieder-Ztg., 1928, [16, 17], 141).

E. LEWKOWITSCH.

Tetrabromide of elæostearic acid. O. MERZ (Farben-Ztg., 1928, 33, 2423—2424).—A review of published work on the bromination of the elæostearic acids. Experiments on the m.p. of mixtures of the tetrabromides of α - and β -elæostearic acids support the view of Rollett (B., 1911, 293) that isomerisation from the α - to the β -form occurs on bromination, the β -form remaining unaffected. E. LEWKOWITSCH.

Soya bean oil. IV. Separation of its glycerides by bromination. K. HASHI (J. Soc. Chem. Ind. Japan, 1928, 31, 117—123).—From the bromination product of soya bean oil three bromides, m.p. 154°, 147°, and 74—75°, respectively, have been separated by means of various solvents. From an examination of the properties of these bromides and their derivatives, the author considers them to be, in the above-mentioned order, linoleodilinolenic bromide, a mixture of oleodilinolenic bromide with dilinoleolinolenin bromide or with linoleodilinolenic bromide, and a mixture of trilinoleic bromide with oleolinoleolinolenic bromide.

Y. NAGAI.

Connexion between analyses and properties of sulphonated oils. F. G. A. ENNA (J. Soc. Leather Trades' Chem., 1928, 12, 204—210).—Sulphonated

castor oil consists almost entirely of one sulphonated fatty acid, whereas fish oils, which contain five different fatty acids, on sulphonation must consist of a variety of products. Castor oil should, theoretically, require 35% of sulphuric acid, but in practice 25—30% is sufficient. Similarly, fish oils should take 60%, but only 15—20% is used. Sulphonated castor oil only makes a leather full and soft, but with sulphonated fish oils the leather is soft, elastic, and well lubricated. A highly sulphonated oil dissolves in water, whilst a moderately sulphonated oil only emulsifies. The presence in fish oils of a large proportion of highly unsaturated fatty acids which can form hydroxy-acids probably explains the superior emulsifying properties of sulphonated fish oils over castor oil sulphonated to the same degree. The methods of analysis used are based on Stiasny and Riess' work (*cf.* B., 1925, 964) and include total fatty matter, unsaponifiable matter, ash, acidity, and total, inorganic, and organic sulphuric acid. D. WOODROFFE.

Sulpholeates in tanning. GERONAZZO.—See XV.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Modern methods of testing paints etc. for weathering and rust-preventive properties. R. KEMPF (Farben-Ztg., 1928, 33, 2359—2361).—Accelerated weathering tests are discussed generally and a bibliography of published work on the subject is given. An alternative to the intensive-cycle procedure is furnished by refined methods of detecting failures in normal exposure tests. Factors influencing outdoor exposure tests are noted and suggestions are offered for controlling these variables so as to increase the value of an outdoor exposure by giving it the reproducibility of a laboratory-accelerated test without the errors inherently induced by the artificial nature of the latter.

S. S. WOOLF.

Accelerated tests of organic protective coatings. P. H. WALKER and E. F. HICKSON (Ind. Eng. Chem., 1928, 20, 591—596).—The apparatus used by the authors for subjecting protective coatings to the destructive agency of artificial light, water, temperature changes, and ozonised air is described. The cycle at present in use comprises refrigeration 2.4%, ozonised air 10.3%, water 19.4%, and light 67.9%, and strict parallelism with outdoor exposures is observed, although no ratio has yet been established. The varying conclusions drawn from the same exposure panel by different competent observers, and by the same observers at different times, emphasise the necessity for refined methods of quantitatively measuring the time and degree of failure of coatings. Suggested procedures are the determination of the permeability of the paint coating to water vapour or air, and the location and measuring of discontinuities in the coating by electrical means. A qualitative method for detecting "pinholes" etc. is described. S. S. WOOLF.

Rapid determination of chromium in lead chromate pigments. G. SIROIS (Chemist-Analyst, 1928, 17, 10).—The pigment (0.2—0.5 g.) is boiled with concentrated hydrochloric acid (20 c.c.), the solution diluted to 60 c.c. with hot water, boiled for 5 min. and

filtered, made slightly alkaline with ammonia, and hydrochloric acid added to dissolve lead hydroxide. The solution is then diluted to 300 c.c., sodium peroxide (3 g.) added, boiled for 20 min., acidified (5 c.c. excess) with hydrochloric acid, cooled quickly to 85°, mixed with 10 c.c. of 20% potassium iodide solution, and immediately and rapidly titrated with thiosulphate.

CHEMICAL ABSTRACTS.

China wood oil [tung oil] in lacquer. I. W. E. FLOOD and D. C. BOOTH [with W. H. BEISLER] (Ind. Eng. Chem., 1928, 20, 609—611).—By introducing raw tung oil into nitrocellulose lacquers opaque films of low tensile strength are obtained. If, however, the tung oil be heated in the presence of ester gum it may be satisfactorily incorporated into lacquers as a "resin component," effecting increased durability, which is still further enhanced by the use of butyl phthalate as plasticiser.

S. S. WOOLF.

Adhesion of [lacquer] films. H. A. GARDNER and A. W. VAN HEUCKEROTH (Ind. Eng. Chem., 1928, 20, 600—601).—The adhesion of various lacquers to black iron, galvanised iron, glass, wood, tin, and aluminium were determined using the Gardner-Parks tensile strength meter (cf. Amer. Paint & Varnish Manufs.' Assoc., 1928, Circ. No. 323). A sheet of silk is embedded in the lacquer coating, which is then pulled off in strips from its iron etc. backing, the instrument indicating tensile strength. The results obtained are interpreted with reference to cohesion of the film, and adhesion of the film to surface and to silk. From the tabulated values it appears that the adhesion of lacquer films to metals increases with the amount of resin up to a certain percentage.

S. S. WOOLF.

Resins from chlorinated cymene. P. H. GROGGINS (Ind. Eng. Chem., 1928, 20, 597—599).—When chlorine is bubbled into *p*-cymene alone or admixed with naphthalene, *o*-dichlorobenzene, etc., in the presence of a metallic catalyst, *e.g.*, aluminium, iron, or zinc, resins insoluble in acids, alkali, and alcohol are obtained. These are not rendered infusible by heat and pressure treatment, and are suitable for use as coating and insulating compositions.

S. S. WOOLF.

Acetone condensation resins. H. A. GARDNER and C. A. KNAUSS (Ind. Eng. Chem., 1928, 20, 599—600).—A preliminary account of the production of durable resins by condensation of acetone with acetaldehyde, paraldehyde, aldol, furfuraldehyde, etc. in the presence of 10% potassium hydroxide solution. Direct addition of rosin and of cellulose esters to the reaction mixtures (using acid in place of alkaline condensation agents) gives resins of satisfactory durability but rather dark colour in comparison with dammar and pale ester gum.

S. S. WOOLF.

PATENTS.

Anti-corrosive paint for iron, steelwork, etc. S. A. McMINN (B.P. 291,486, 28.1.27).—Dehydrated oil-gas tar is mixed with aluminium powder or similar metallic substance.

F. G. CROSSE.

Production of lead minium [red lead] having a high peroxide content. CONSORT. ELECTRO-CHIM. DE FRANCE, Assees. of J. J. TARDAN (B.P. 283,898, 18.1.28.

Fr., 19.1.27).—Lead converted into a spongy or pasty condition, *e.g.*, by electrolytic means, is dried slowly at a temperature rising progressively to 200°, *e.g.*, by conveying it through a shallow chamber in which it is dried by a current of hot air, before it is oxidised by roasting in a furnace of the rotating and rocking type.

L. A. COLES.

Removal of drawing ink. F. N. STEIGLEDER (U.S.P. 1,672,790, 6.5.28. Appl., 20.7.25).—An ink eradicator consists of the reaction products of an alkali hypochlorite, chlorine water, and carbon dioxide in water.

H. ROYAL-DAWSON.

Manufacture of butaldehyde-phenol resins. C. F. SCHRIMPE, Assr. to BAKELITE CORP. (U.S.P. 1,667,872, 1.5.28. Appl., 27.9.22. Renewed 26.9.27).—Butaldehyde is heated with a phenol, preferably in the presence of an acid condensing agent, such as hydrochloric acid. The resulting resin is not converted into an inert, infusible resin by heating unless compounds containing a reactive methylene group, such as formaldehyde and hexamethylenetetramine, are added.

F. G. CLARKE.

Containers for corrosive chemicals (B.P. 283,868). **Titanium pigments** (B.P. 290,683-4).—See VII. **Protective coverings for aluminium** (U.S.P. 1,672,230).—See X.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Seasonal variations in the composition of the latex of *Hevea brasiliensis*. N. RAE (Analyst, 53, 330—334).—Monthly analyses of the latex of *Hevea brasiliensis* and its ash were made from Colombo trees that were being heavily tapped. Variations in content of resin, water, rubber, and in *p_H* value showed no relation to rainfall; a relationship between the conductivity and ash of the latex was established, and a general correspondence between proteins in the latex and ash and therefore the mineral salts. Nitrogen, potash, and phosphoric acid were at a minimum in August, when seed fall occurs; they increased till February, and then decreased rapidly till May, followed by a slight increase to July and a drop to August. The large fall in March coincides with leaf fall and renewal of leaf, and it is suggested that the latex acts as a food reserve to be used up when leaf and seed growth are at a maximum.

D. G. HEWER.

Carbon blacks and their use in rubber. I. Comparative properties of blacks and tests in uncured rubber. N. GOODWIN and C. R. PARK (Ind. Eng. Chem., 1928, 20, 621—627).—A lamp black, two gas blacks produced by the channel process for use in rubber and varnish respectively, and two blacks obtained from natural gas by thermal decomposition and by incomplete combustion at high temperature are examined as to sp. gr., bulkiness, particle size (in rubber), X-ray pattern, tinting strength, colour undertone, particle shape, extractable matter, adsorptive capacity for iodine or methylene-blue, moisture content, oil absorption, sedimentation in various media, maximum proportion introducible into rubber, and behaviour of rubber mixings, containing the respective blacks, towards milling, solvents, and blooming.

D. F. TWISS.

PATENTS.

Production of rubber articles. K. MEDGYES (B.P. 277,928, 4.3.27. Hung., 25.9.26).—The use of the customary dusting powders to prevent inadvertent adhesion between plies of sheet rubber has the effect of greatly reducing the strength of the seams produced when such sheets are formed into articles by joining their edges with pressure. Almost the full strength of the rubber in such welded seams can be obtained by using for dusting purposes substances which lose their anti-cohesive effect during subsequent heating or vulcanisation. Examples are naphthalene, hexamethylenetetramine, salts of fatty acids with zinc or lead, alcoholic solution of acetanilide, and aqueous suspension of thiocarbanilide. D. F. TWISS.

Manufacture of reinforced rubber articles. W. B. WESCOTT, Assr. to RUBBER LATEX RES. CORP. (U.S.P. 1,671,914, 29.5.28. Appl., 25.11.25).—By passage through a bath of latex, fibrous material is impregnated with latex and given a surface coating of rubber. After coagulation of the absorbed latex, compression is applied to remove water, and soluble impurities are leached out by washing. The material is then dried in a vacuum at about 65°, and compacted by pressure while warm. D. F. TWISS.

XV.—LEATHER; GLUE.

Chrome tanning. XVIII. Analytical figures for [chrome-tanned] leather. D. BURTON and H. CHARLTON (J. Soc. Leather Trades' Chem., 1928, 12, 210—216).—The sum of the percentage amounts of moisture, grease, hide substance, water-soluble matter, and ash as determined by the analysis of chrome-tanned leather is frequently much less than 100. The grease determination by extraction of the leather with petroleum spirit is inaccurate. Up to 1.5% of skin fat may be retained by the leather. Contrary to Stiasny and Riess' results (cf. B., 1925, 964), the authors found that sulphonated oils are extracted by petroleum spirit. Wool fat soaps and neutral soaps of the alkali metals are not readily soluble in petroleum spirit, and only 83% of rosin dissolves in the solvent. Again, extraction with petroleum spirit of a leather containing sulphur removes some of the sulphur. Low results are obtained for the water-soluble matter owing to the difficulty of wetting back chrome-tanned leathers. It is shown that the grease in a waterproofed chrome-tanned leather causes low results for the nitrogen determination, and such leather should be degreased before Kjeldahling. In order to determine the specific gravity of a leather, a burette is half filled with mercury and the reading noted. Weighed strips of the leather, 4 in. long, are inserted until the mercury rises nearly to the zero mark, the leather being kept below the surface of the mercury with a pin; the reading is again taken. The difference in the readings will give the volume, from which the density can be calculated. D. WOODROFFE.

Depilation of skins by means of alkaline solutions. R. H. MARRIOTT (J. Soc. Leather Trades' Chem., 1928, 12, 216—234).—The effect of various oxidising and reducing agents in suspensions of calcium hydroxide was tried on sun-dried goat skin, which had been previously soaked back in water. After 19 hrs. the order of

depilatory power was: lime and potassium cyanide, lime and sodium sulphite, lime and ferrous sulphate, old lime from ox hide, old lime from ox hair, fresh lime suspensions, lime and ferric sulphate, lime and chloroform. These results indicate that the unhairing properties of lime liquors are mainly due to the reduction of the keratin molecule, probably of the —S—S— linking in cystine, since cyanides, sulphites, and sulphides, which are known to reduce this grouping, markedly increase the rate of unhairing. Air-free lime liquors loosened the hair more quickly than an ordinary lime liquor, which fact supports the above theory. When 0.5% of potassium cyanide was added to a lime liquor, and the solution tried on goat skin, the hair was rotted in 24 hrs.; similarly, 1% of sodium sulphite just loosened the hair, 1% of calcium sulphite showed less loosening, but was more affected than the lime liquor alone; 1% of ferric chloride and potassium persulphate respectively, prevented any loosening of the hair. The addition of sodium sulphite to a lime liquor gives a much higher concentration of sulphite ion than the addition of calcium sulphite. The presence or absence of suspended matter in the lime liquors did not affect the unhairing properties. It was found that, by soaking the skins in a solution containing sodium sulphite, bisulphite, or sulphurous acid, the loosening of the hair was accelerated in the subsequent liming process. The effect of sulphites was greatest in alkaline solution. A 0.1N-sodium hydroxide solution containing 5 g. of sodium sulphite crystals per 400 c.c. of solution was used for soaking sun-dried goat skin for 2 days, after which the latter was transferred to a 1% solution of sodium sulphide crystals for 6 hrs. The hair roots came out easily, leaving the grain white and clean. The hair was not damaged. D. WOODROFFE.

Effect of egg yolk on the distribution of oil in chrome calf leather. H. B. MERRILL (Ind. Eng. Chem., 1928, 20, 654—656).—Samples of wet, neutralised, chrome-tanned calfskins were fat-liquored with sulphonated neatsfoot oil and egg yolk in various proportions respectively, then rinsed, partly dried, split into five layers, and the fat and moisture determined in each layer. The use of sulphonated oil alone caused the fat content of the skin immediately below the grain to be higher. The addition of egg caused the flesh side to absorb twice as much oil as the grain. As the egg content of the fat-liquor was increased, the ratio of the fat content of the grain to that of the flesh decreased. Further, when the total amount of fat used was kept constant and no egg was used, the grain layer contained 25% more fat than the flesh. As the ratio of egg to oil was increased the ratio of fat in the grain to fat in the flesh fell, until with a pure egg fat-liquor the grain contained only two fifths as much fat as the flesh. The addition of fresh egg white to a fat-liquor increased the amount of fat taken up by the flesh side much more than egg yolk or whole egg. The addition of egg to a sulphonated oil emulsion increased the size of the droplets considerably, which may account for the diminished penetration of the grain layer. D. WOODROFFE.

Analytical control of sulpholeates used in tanning. II. Sulphoricinoleates. M. GERONAZZO (Boll. Uff.

Staz. Sperim. Ind. Pelli, 1927, 5, 416—420).—Good sulphuricinoleate should be clear; of straw to golden-yellow colour, and homogeneous, the presence of suspended or dissolved matter indicating watering or faulty manufacture. Its reaction may vary according to the methods of washing and neutralisation followed; alkaline preparations are unstable, and different acidities are desirable for different purposes. Determination of the reaction towards test-papers might well be replaced by that of the acid value, which agrees best with the reaction to phenolphthalein. Whether ammonia or soda or both have been used in neutralising the excess of sulphuric acid used in the manufacture may be determined by the ordinary methods. The emulsive power may be measured by shaking 2 c.c. of the sulphuricinoleate for about 2 min. with 20 c.c. of water, the quality of the product being related directly to the duration of the emulsion and to the emulsive capacity, i.e., the property of retaining a definite quantity of another fatty substance in homogeneous emulsion. Addition of ammonia, drop by drop, to the emulsion should result in a clear liquid, turbidity indicating defective manufacture or the presence of mineral or vaseline oil etc. The titre of the sulphuricinoleate may be determined by shaking 25 c.c. of the material in a graduated cylinder with 25 c.c. of dilute sulphuric acid (from 30 c.c. of acid, d 1.835, and 70 c.c. of water) for about 1 min., and leaving at rest for about 12 hrs.; the number of c.c. of the upper oily layer, when multiplied by 4, gives the commercial titre of the product. The presence of iron in the sulphuricinoleate may be detected by treating the lower liquid layer formed in the previous test with a few drops of 5% aqueous potassium ferrocyanide; not more than a small proportion of iron should be present. T. H. POPE.

Fixation of aluminium by hide substance. A. W. THOMAS and M. W. KELLY (Ind. Eng. Chem., 1928, 20, 628—632).—Portions of defatted hide powder were treated with a large excess of solutions of aluminium sulphate and chloride for 24 hrs., well washed, air-dried, ashed, and weighed. It was found that the p_H value of the solutions could be varied from 3.2 to 3.8 for the sulphate and from 3.4 to 4.0 for the chloride. Very little aluminium was fixed from the normal salts, but the amount fixed by the powder increased with increasing p_H value, while at the same time the tannage improved. Greater fixation of aluminium took place from aluminium chloride solutions up to p_H 3.65; beyond this point the salt was precipitated. Aluminium sulphate was the better tanning agent, because it could be rendered more basic without precipitating the hydroxide. The maximum amount of alumina fixed was 3.5 g. (as Al_2O_3) per 100 g. of hide substance. The optimum concentration of the aluminium salt for the fixation of aluminium was 0.2*N*. The time required for complete tannage was only a few hours. Additions of sodium chloride or sulphate, especially of the latter, to the tanning solutions diminished the amounts of aluminium fixed by the hide substance.

D. WOODROFFE.

Fixation of iron by hide substance. A. W. THOMAS and M. W. KELLY (Ind. Eng. Chem., 1928,

20, 632—634).—Portions of defatted hide powder were treated with solutions of ferric sulphate of different basicities, washed free from iron and sulphates, dried, ashed, and weighed. Iron was fixed from solutions of normal ferric sulphate, but the amount increased as the basicity increased. The tanning action was very rapid—1 hr. with normal sulphate solutions and 6 hrs. with basic solutions; 7 g. of iron oxide (as Fe_2O_3) combined with 100 g. of hide substance, whilst the minimum quantity required to form an iron collagenate would be 7.1 g. Sodium chloride diminished the amount of iron fixed by the powder. Sodium sulphate showed less inhibitory effect in basic solutions, and small quantities favoured the fixation of iron from normal ferric sulphate solutions, thus differing from its effect on chrome and alum tanning. D. WOODROFFE.

Sulphonated oils. ENNA.—See XII. **Tannery waste** (U.S.P. 1,672,586).—See XXIII.

XVI.—AGRICULTURE.

Soil adsorption. D. J. HISSINK [with J. VAN DER SPEK, A. and M. DEKKER, and H. OOSTERVELD] (Proc. Internat. Soc. Soil Sci. [Suppl.], 1928, 1, 4—42).—Comparison is made of the methods for determining exchangeable bases in soils. The determination of the value S in chalk-free soils by leaching with neutral salts presents no difficulty, but in carbonated soils some calcium carbonate is extracted during leaching. In successive leachings with *N*-sodium chloride solution (Hissink) this amount is practically constant. This is not the case in the Kelley method using ammonium chloride. Further, during the distillation of adsorbed ammonia from soil (Kelley) some ammonia results from the decomposition of organic matter. Values of S are usually lower by Kelley's method than by Hissink's. In acid soils exchange of hydrogen ions for ammonium ions tends to increase the Kelley values. The removal of ammonia from the soil complex in colloidal solutions during washing tends to reduce the value of S (Kelley) in clays and humus soils. In the Bobko-Askinasi process similar factors operate, and, in addition, more hydrogen ions in soils are replaced by barium ions than by ammonium ions, and less barium is removed during the washing operation. The value of S (Bobko-Askinasi) is higher than that of S (Hissink) for very acid soils, and lower for slightly acid or alkaline ones. In Kappen's method for determining hydrolytic acidity, the factor 1.5 used to calculate lime requirement is applicable to mineral soils, but should approach the value 2 where appreciable quantities of humus exist. Methods for determining lime requirements involving treatment of soil with lime, chalk, or bicarbonate solution yield values largely influenced by the relative proportions of soil, reagent, and water. The p_H value of soils in aqueous suspension, as determined by the Büllmann electrode, are greater than when a *N*-potassium chloride or *N*-calcium acetate suspension is used. The term "adsorption capacity" (Gedroiz) is based on the adsorptive value of the soil for barium and is not representative of the total adsorption capacity, since hydrogen ions in soil are not completely replaceable by barium ions. In order satisfactorily to deter-

mine the lime requirement of a soil and to include therein a correct representation of the physical and chemical effects of calcium salts on the mineral and organic complexes, it is recommended to determine chalk, humus, and clay (diam. $<16\mu$) contents, reaction, state of saturation, and adsorption potential calculated per g. of soil and per 100 g. of adsorbing agents (clay and humus), and hence the lime factor.

A. G. POLLARD.

Absorption phenomena in acid soils. W. HILLKOWITZ (Z. Pflanz. Düng., 1928, A11, 229—264).—Treatment of soil with increasing amounts of hydrochloric acid showed that no decomposition of the zeolite complex occurs with acids weaker than 0.1N. Changes in the adsorptive power of acidified soils for calcium were irregular, but generally speaking those originally having considerable amounts of exchangeable calcium adsorbed but little more, and those having much exchangeable aluminium possessed strong adsorptive power for calcium. Increased exchange acidity in soils is accompanied by greater adsorptive power for phosphate applied as calcium dihydrogen phosphate. Adsorption from ammonium phosphate follows different lines. Similar phenomena were observed in experiments with permutite. The relative "adsorptive energy" of the zeolite-humus complex for different cations is not always in accord with Hissink's views. Neutralisation of previously acidified soils and permutites does not completely restore their original adsorptive powers. Simplified methods for determining exchangeable calcium and the total exchangeable bases in soils are described and their limitations discussed.

A. G. POLLARD.

Acid properties of artificial and soil permutites. W. U. BEHRENS (Z. Pflanz. Düng., 1928, A11, 281—287).—Permutites from which bases are removed function as acids and can be titrated as such. The p_H values of partially neutralised "permutite acids" depend upon the proportion of electrolyte present. "Permutite acids" are comparable in strength with carbonic acid, the soil acids being slightly stronger than artificial ones. The results of treating permutites with neutral salt solutions are explicable on the basis of the law of mass action.

A. G. POLLARD.

Titration curves and buffer actions of soils. F. TERLIKOWSKI and T. WLOCZEWSKI (Rocz. Nauk Rolniczych i Lesnych, 1925, 13, 602—603; Bied. Zentr., 1928, 57, 150—151).—The buffering properties of fifteen soils, including various horizons of those soils, were examined, on the ground that such properties vary in significance according to the horizon. The reaction of these soils with hydrochloric and sulphuric acids was determined at C_H values ranging from 2.5×10^{-4} to 2.25×10^{-2} g./litre. The reaction with superphosphate containing free acid, in quantities equivalent to the pure acid solutions and also similar to those used in agricultural practice, was also investigated. 10 g. of soil were mixed with 20 c.c. of acid and maintained at 18° for 48—96 hrs.; the p_H value was then determined potentiometrically. There were examined seven humus horizons, of partially podsolised, podsol, and meadow soils; three eluvial horizons; three illuvial horizons of the same soil; and also two C-horizons of sandy forest soils. The chief

findings were as follows: All soils, independent of their initial reaction, showed buffer action, even pure sand formations. As a measure of the buffering properties can be taken the area, bounded on the one side by the theoretical titration curve and on the other by the experimental titration curve, taking into account the initial reaction. In the soil profile, the least buffer action is found in the eluvial horizon; this is explained by the soil-building process in this horizon. In this connexion are often found significant buffering properties in illuvial horizons, which are produced therein by the products of leaching of the A_1 - and A_2 -horizons. The A_2 -horizon particularly has the highest buffer action, indicating therein the rôle of humic substances. No distinction in buffering properties could be drawn between forest and arable soils. Greater buffer action is displayed by soils towards free acids than towards superphosphate. The use of the latter in quantities corresponding to those used in agricultural practice caused little or no alteration in soil reaction.

H. J. G. HINES.

Liming [of soils]. A. GEHRING (Z. Pflanz. Düng., 1928, B7, 249—264).—Important points in liming practice are discussed. The form of lime used, the manner and period of application in the crop rotation, and the conjoint use of fertilisers are considered.

A. G. POLLARD.

Influence of soil structure on fertility. A. ACHROMEIKO (Z. Pflanz. Düng., 1928, A11, 36—50).—Comparative cropping experiments are described in soils in natural, crumbly, and powdery conditions. If nutrient and water supplies are similar, the crop yield is not appreciably affected, but differences in "non-capillary porosity" markedly influence germination, low porosity being coincident with poor germination. A loose, as compared with a compacted, soil surface favours germination, but does not affect the total crop yield to a marked degree.

A. G. POLLARD.

Texture and structure of soils as influenced by chemical reagents. G. J. BOUYOUCOS (J. Amer. Soc. Agron., 1927, 19, 788—797).—The behaviour of various soils on treatment with salts, acid, alkali, or sugar was studied.

CHEMICAL ABSTRACTS.

Chemical analysis of soils and the molecular ratios. K. UTESCHER (Z. Pflanz. Düng., 1928, A11, 265—281).—The methods of the Preussen Geologisches Landes-anstalt for the extraction of soils with hydrochloric acid are discussed, and the molecular ratios thus obtained are compared with soil acidity measurements. A more concentrated acid is recommended for use. Details for a complete chemical analysis of soil are appended.

A. G. POLLARD.

Determination of magnesium in soils. E. DUPONT (Ann. Sci. agron. Franç., 1927, 43, 458—470; Chem. Zentr., 1927, II, 1392).—I. The soil extract (100 c.c. = 10 g. of soil) is neutralised with ammonia and the mixture shaken with 10 c.c. of acetic acid to dissolve the iron precipitate. The mixture is diluted to 300 c.c. and treated with the calculated amount of 4.7% ammonium oxalate solution on the basis of oxalic acid = $1.3\text{CaCO}_3 + 7\text{Fe} + 2$. After 3 hrs. the liquid is tested to show the complete precipitation of calcium, a little kiesguhr is added, and the whole diluted

to 500 c.c. and filtered. To 301.7 c.c. of the filtrate a few drops of phenolphthalein are added, followed by 50 c.c. of a neutral citrate solution (40% citric acid), 15 c.c. of ammonium diphosphate solution (15% P_2O_5), and ammonia (d 1.23) till alkaline. The magnesium precipitate is stirred up and 60 c.c. of ammonia solution are added. After 24 hrs. the solution is filtered, the precipitate washed and dissolved from the filter with warm nitric acid (5%), and the solution evaporated to dryness in a platinum dish; the residue is ignited and weighed as magnesium pyrophosphate.

II. To 90.5 c.c. of soil extract (9.05 g. of soil) without preliminary treatment are added 50 c.c. of citrate solution, 10 c.c. of phosphate solution, and ammonia till alkaline to phenolphthalein and the solution precipitated as before. After filtration the precipitate is digested with 10 c.c. of citrate solution and 40 c.c. of ammonia for 2—3 min., filtered, washed free from citrate, and treated as described above.

A. G. POLLARD.

Mechanical analysis [of soils], national and international. B. A. KEEN (Proc. Internat. Soc. Soil Sci. [Suppl.], 1928, 1, 43—49).—The official English (1925) method for the mechanical analysis of soils as now modified (Dec., 1927) brings it into line with the method prescribed by the International Society with the sole exception that values are expressed as percentages of air-dried soil. A plea is made for general agreement among other countries for the adoption of the International method or one the values of which can be converted into the International scale by a simple calculation.

A. G. POLLARD.

Mechanical analysis of soils. III. A new pipette. M. KÖHN (Z. Pflanz. Düng., 1928, A11, 50—54. Cf. B., 1927, 855; 1928, 27).—A special pipette for the new method for the mechanical analysis of soils is described. The larger particles are determined by elutriation methods. Errors in the Atterberg sedimentation process are illustrated.

A. G. POLLARD.

Mitscherlich's method for determining the fertiliser requirement of soils. M. GERLACH, E. GÜNTHER, and C. SEIDEL (Z. Pflanz. Düng., 1928, A11, 1—35).—The Mitscherlich hypothesis of growth factors is not in accord with experimental results. Mitscherlich's equation represents only the nutrients absorbed by the growing plants and not the total soil nutrient. The value of the growth factor is constant only when the water and nutrient supplies to the plant are maintained at a constant value and the progressive increases in crop yield are generally applicable to the calculation of nutrient values. Only the actual experimental figures in the Mitscherlich process are utilisable, and these accomplish no more than a well-conducted field trial.

A. G. POLLARD.

Action of nitrogenous fertilisers on the quality of potatoes for "seed" and on the composition of potatoes grown on four different soils. E. KRÜGER (Landw. Jahrb., 1927, 66, 781—843; Bied. Zentr., 1928, 57, 156—158).—The quality of seed potatoes was distinctly influenced by the application of different nitrogenous fertilisers in increasing amounts. The soils themselves were an important factor also, moorland

soils giving the best and heavy calcareous soils the worst types of seed potatoes. The heavier nitrogenous dressings gave increased yields on the moorland and sandy soils without diminution in quality, but the increase in yield on the heavier soils was accompanied by a lowering of quality. Ammonium sulphate and urea were more effective than sodium and calcium nitrates or calcium cyanamide. Analyses of the differently fertilised crops revealed differences in the total nitrogen contents and in the distribution of the nitrogen. A low ratio of amino-acid to total nitrogen during the winter resting period is an indication of good seed quality.

H. J. G. HINES.

Factors determining the availability to plants of difficultly soluble calcium phosphates. W. W. BUTKEWITSCH (Landw. Jahrb., 1927, 66, 947—992; Bied. Zentr., 1928, 57, 154—156).—The type of culture vessel used in this work consisted essentially of a large glass cylinder in which was placed a narrower glass cylinder. In the outer compartment was a complete nutrient solution omitting phosphate. The inner cylinder contained sand resting on a porous plate, leaving a small space below. A siphon and air tube reached this space. Seedlings germinated in pure water were placed with their roots partially in the sand and partially in the nutrient solution. In order to study the behaviour of various phosphates, these were mixed with the sand. The effect of various solutions percolating the sand on the solubility of the phosphates could be estimated by withdrawing the percolated solution through the siphon. It was shown that the availability of the insoluble calcium phosphates depends in the first place on the properties of the soil solution, particularly on the concentrations of calcium, bicarbonate, and hydrogen ions. The utilisation of these phosphates by plants does not depend entirely on their solubility, and is influenced by the buffer system in the soil solution. The separate and combined influences of various ions on the solubility of the phosphates and on their utilisation by plants are described.

H. J. G. HINES.

Chemical treatments for controlling the growth of buds of plants. F. E. DENNY (Ind. Eng. Chem., 1928, 20, 578—581).—Early germination of recently harvested potatoes may be brought about by treating them with a solution of sodium thiocyanate or by exposing them to vapours of ethylene chlorohydrin, gains of 2 weeks to 2 months in the time required to cause sprouting having been obtained. With chlorohydrin the temperature should be below 32°, otherwise the potatoes are injured. The development of buds in certain woody plants such as lilac, crab-apple, deutzia, etc. may be accelerated to a similar extent by preliminary treatment with chlorohydrin, ethylene dichloride, furfuraldehyde, ethyl bromide, or vinyl chloride, the type and rate of growth depending on the concentration of the chemical used. Chlorohydrin is also effective with gladiolus bulblets. When growing potatoes for seed purposes, the tuber may be advantageously soaked in a solution of thiourea, which induces multiple bud development.

F. R. ENNOS.

Acetic acid as a soil disinfectant. W. L. DORAN (J. Agric. Res., 1928, 36, 269—280).—The application

of 1–1.2% acetic acid to soil is found to protect tobacco against black root rot, brown root rot, and bed rot, and to protect cucumber, tomato, lettuce, and white spruce from injury by damping off during or after germination. The time which must elapse between the application of the acetic acid and the planting of seed has been found to vary with the species of plant and the variety of soil. E. A. LUNT.

Soil treatments and seasonal chemical changes in the sour cherry. A. G. ANDERSON and H. D. HOOKER (Missouri Agric. Exp. Sta. Res. Bull., 1927, No. 108, 27 pp.).—Spurs and shoots of sour cherry trees on plots (a) tilled, (b) in sod, (c) in sod with spring applications of sodium nitrate, were analysed. Conversion of starch into sugar takes place during the winter, and re-synthesis of starch in March. There is marked increase of nitrogen in spurs and shoots at the beginning of active spring growth. Carbohydrate is maximal in March, May (end), and September. Maximum fruit bud formation is associated with maximum carbohydrate accumulation by the end of June.

CHEMICAL ABSTRACTS.

Concord grape: fruiting habits of the vine. A. L. SCHRADER (Maryland Agric. Exp. Sta. Bull., 1926, No. 286, 62–118).—In the dormant season the roots of a 1-year vine contained 75% of the total carbohydrates and 80% of the total nitrogen. Nodes 1, 2, and 3 are low in starch, nitrogen, and sugar; 4 and 5 are high in starch, medium in sugar and nitrogen; 6, 7, 8, and 9 are medium in starch and high in sugars and nitrogen.

CHEMICAL ABSTRACTS.

Influence of environmental conditions on the activities of cellulose-decomposing organisms in the soil. R. J. DUBOS (Ecology, 1928, 9, 12–27).—The moisture content is important; the abundance or otherwise of the nitrogen supply does not appreciably influence the nature of the organisms concerned. In acid soils under aerobic conditions the decomposition is effected only by fungi.

CHEMICAL ABSTRACTS.

Maple sap and its improvement. E. HART (Ind. Eng. Chem., 1928, 20, 581).—Maple sap normally contains 2–3% of sugar. An attempt is being made to increase this yield by seed selection, in order to allow of a more economical production of maple sugar.

F. R. ENNOS.

Fertiliser tests with flue-cured tobacco. E. G. MOSS, J. E. McMURTRY, W. M. LUNN, and J. M. CARR (U.S. Dep. Agric. Tech. Bull., 1927, No. 12, 1–58).

Salt requirement of tobacco grown in sand cultures. H. LIN (Maryland Agric. Exp. Sta. Bull., 1926, No. 288, 132–153).

PATENTS.

Production of fertilisers from vegetable refuse. J. JACKSON (B.P. 291,514, 2.3.27).—Waste vegetable matter is pulverised and agitated with water to separate extraneous matter. The wet material is collected, e.g., on a continuous band strainer, pressed between rollers, dried, and powdered. The press juice may be collected and treated to recover alcohol etc. Suitable machinery is described.

A. G. POLLARD.

Treatment of phosphate rock. G. T. HARNED, Assr. to PHOSPHATE MINING CO. (U.S.P. 1,671,765,

29.5.28. Appl., 20.7.27).—Calcined rock, while hot, is mixed with a predetermined proportion of wet rock, thus using the sensible heat to dry and increase the calcium phosphate content of the latter.

H. ROYAL-DAWSON.

XVII.—SUGARS; STARCHES; GUMS.

Effect of animal char on the invert sugar in products of the sugar-refining industry. M. I. NAKHMANOVICH and I. F. ZELIGMAN (Nauch. Zapiski, 1927, 5, 81–87).—The interaction of animal char with inverted sucrose causes a lowering of the levorotation, due to adsorption or to chemical action catalysed by the carbon. With low-purity products the levorotation is increased on account of adsorption of dextrorotatory substances.

CHEMICAL ABSTRACTS.

Maple sap. HART.—See XVI.

XVIII.—FERMENTATION INDUSTRIES.

Analytical characterisation of fermentation vinegar. L. A. JANKE and F. POPBERGER (Deuts. Essigind., 1927, 31, 257–259, 265–267; Chem. Zentr., 1927, II, 1410).—Fermentation vinegar may be distinguished from dilute spirit vinegar by the determination of its iodine value (mg. of iodine required to react with that amount of the original substance which contains 10 g. of acetic acid), which at 40° should be at least 50. A sample (about 40 c.c.) of the vinegar is evaporated to approximately one quarter of its volume and made up again to its original volume. To 10 c.c. of this solution are added 40 c.c. of 0.01N-iodine and 8 c.c. of 2N-sodium hydroxide more than are required for neutralisation, and the whole is warmed to 40°. After 2 hrs. 2N-hydrochloric acid is added (4 c.c. more than the volume of sodium hydroxide previously required), and 8 min. later the liquid is titrated with 0.01N-thiosulphate. Admixture of the fermentation vinegar cannot be determined unless the iodine value is known. Filtration of the fermentation vinegar through wood charcoal lowers the iodine value owing to the adsorption of a portion of the iodine-reacting constituents, which appear to be related to vitamin-D.

C. RANKEN.

Citric acid fermentation. M. S. FILOSOFOV and V. E. MALINOVSKI (Nauch. Zapiski, 1928, 5, 235–239).—In 20 days *Citromyces* produced citric acid equal to 17.1% of the sugar in the modified Henneberg medium; other moulds were also examined.

CHEMICAL ABSTRACTS.

Fermentation of toddy and the micro-organisms producing it. M. DAMODARAN (J. Indian Inst. Sci., 1928, 11A, 63–74).—Yeasts and bacteria concerned in the fermentation of toddy are isolated and described. These appear common to all samples of toddy irrespective of origin and locality. The bacteria (sp. *Acetobacter*) do not markedly influence alcohol production, but are responsible for turbidity, unpleasant odour, and the production of acetic acid. The elimination of these bacteria by heat-treatment is suggested, the death point of the bacteria being 38–39° as against 52–53° for the yeasts. Vinegar production by mere prolonged storage of toddy is uneconomical. A. G. POLLARD.

PATENTS.

Manufacture of yeast. H. C. and J. P. H. JANSEN (B.P. 291,127, 3.5.27).—The solution of the nutrients is introduced in the form of a spray into the air or other gas used for aerating the wort, and the mixture passed into the seeded wort. Should ammonia or other volatile liquid be used in the process, the air may be passed through such liquid on its way to the aeration vessel. It is claimed that with this arrangement the yield of yeast is increased (cf. B.P. 270,770; B., 1927, 568).
C. RANKEN.

Manufacture of yeast. J. Y. JOHNSON. From H. BÜCHER (B.P. 291,146, 24.2.27).—In a continuous process, a sugar solution containing mineral yeast food flows slowly with seeded yeast from a storage vessel into a propagating vessel provided with suitable aerating devices. Nutrients and sugar are added at points to ensure a suitable concentration of such substances being maintained. The liquid, separated by a centrifuge from the yeast, is pumped back to the storage vessel and used again after the addition of fresh nutrient substances. After repeated use of such wort, it may be sterilised by heat or purified by passing over activated charcoal. The discharged air, automatically freed from foam, flows into an absorption vessel where the alcohol and aldehyde are absorbed by a suitable liquid.
C. RANKEN.

Treatment of yeast. W. SCOTT (B.P. 291,135, 22.2.27).—Yeast, after fermentation, is skimmed or sucked into an enclosed vessel provided with a rouser, which is adapted to circulate the liquid portion of the skimmings until the desired degree of maturity of the yeast is reached. Subsequent to the reconditioning of the yeast, the bulk of the liquid is allowed to separate by gravitation and filtered separately from the yeast. By means of a jacket or a coil the contents of the enclosed vessel may be brought to any desired temperature.
C. RANKEN.

XIX.—FOODS.

Tallowiness or rancidity in grain products. M. S. FINE and A. G. OLSEN (Ind. Eng. Chem., 1928, 20, 652—654).—Grain products such as entire-wheat flour, white patent flour, maize, and wheat germ containing less than 2% of moisture develop a tallowy odour relatively rapidly, especially at higher temperatures. With 5% of moisture the change is considerably delayed, this "protective" effect increasing with rise in the moisture content, so that samples with 10—12% of moisture may be preserved fresh for 3 years. Suitable addition of moisture can also exert a "curative" effect in materials of initially low moisture which have developed tallowiness. As little as 0.25% and 0.5% of glycerol exerts a protective action on baked grain products for 2 and 3 years respectively.
F. R. ENNOS.

Analytical detection of bleaching of wheat flour. H. JØRGENSEN (Res. Bage- og Mellaboratoriet, Copenhagen, 1928, 70 pp.).—The agents at present used in the bleaching of flour are nitrogen peroxide, chlorine, nitrogen trichloride, and benzoyl peroxide, and methods of using them are reviewed. A modification of Winton's gasoline colour value test (U.S. Dept. Agric., Bull. No.

137, 1911) in which the colour given to gasoline by the carotin in the flour is compared with that of a 0.05% solution of potassium chromate in water is described. The lowest colour value which 66 samples of recently-milled unbleached flour were found to have was 110, compared with the standard chromate solution as 100. Since the colour value decreases on storage owing to natural bleaching, test samples should always be drawn from the core of the bag. The lowest colour value unbleached flour may be expected to have in practice is 95; if it falls below 90 the flour must be classified as "bleached." Nitrogen peroxide bleaching may be detected, and the nitrous acid in the flour determined, by the Griess-Ilosvay test, which depends on the red coloration given by sulphanilic acid and α -naphthylamine together in presence of nitrous acid. If a sample of flour contains more than 40×10^{-5} g. of nitrite nitrogen per kg., treatment of the flour with nitrogen peroxide or with some other agent that leaves a residual nitrous acid content may be suspected. Chlorine bleaching may be detected by the presence of chlorine in the fat. For this purpose the Beilstein flame test is unreliable, but incineration of the saponified fat and titration of the chloride with 0.02N-silver nitrate solution and ammonium thiocyanate solution is effective. If 15 mg. or more of chlorine are found in the fat from 1 kg. of flour, chlorine bleaching may be assumed. Bleaching with nitrogen trichloride may be inferred from the extent of the chlorine present in the fat. Benzoyl peroxide bleaching cannot be detected after two days, as the peroxide disappears and the residual benzoic acid is only present in the proportion of 0.02 pt. per thousand. Rothenfusser's test, in which *p*-diaminodiphenylamine sulphate is the reagent, can, however, be used to detect undecomposed benzoyl peroxide.

W. J. BOYD.

Relation of milk solids-not-fat to overrun and quality of ice cream. P. S. LUCAS and W. J. ROBERTS (Mich. Agric. Exp. Sta. Tech. Bull., 1927, No. 86, 1—16).—The apparent viscosity of an aged ice cream mix is increased by increasing the milk solids-not-fat content; these have little effect on the overrun, but increase the density and lower the f.p.

CHEMICAL ABSTRACTS.

Glycerin in cream. A. F. LERRIGO (Analyst, 1928, 53, 335—336).—Glycerin in cream may be detected during and immediately after determination of total solids. Fumes may be observed on removing the dish from the oven after $\frac{1}{2}$ hr. with as little as 1% of glycerin. After weighing, further heating will produce a slight rise in weight with genuine cream, but a continued loss will occur if glycerin is present, when darkening of the solids after 2 hrs.' heating is also much more marked.

D. G. HEWER.

Tests for reconstituted cream. F. W. RICHARDSON (Analyst, 1928, 53, 334—335).—5 c.c. or 5 g. of cream are shaken with a mixture of equal parts of benzene and methylated spirit (60 o.p.) and the mixture is centrifuged. Fresh cream even when it has become sour will remain as an emulsion, but reconstituted cream will throw up its butter fat; after strong centrifuging in the latter case three layers will be present, and in the former only the aqueous layer and the upper opaque stratum.

Reconstituted cream submitted to a sterilising process will give no reductase action with methylene-blue.

D. G. HEWER.

Determination of butter in margarine. L. V. COCKS and E. NIGHTINGALE (Analyst, 1928, 53, 322—327).—Deviations in results for Reichert-Meissl-Polenske-Kirschner determinations were obtained in spite of strict attention to details (cf. B., 1911, 975). Low Kirschner values resulted from the use of inferior quality silver nitrate, and sulphuric acid was found in the Kirschner distillate when the asbestos shield did not perfectly fit the bottom of the distillation flask, owing to volatilisation by overheating through the annular spaces; this is avoided if uralite plate is used instead of asbestos, with a central 5 cm. diam. bevelled hole, and the final joint made with asbestos paper. Sulphuric acid did not volatilise in the presence of the fatty acids on the walls of the flask, but with the larger excess of acid in the Kirschner determination it distilled over. Interpretation of results necessitates the knowledge of the Kirschner value of the original butter fat, since this may vary from at least 19 to 27. Assuming an original value of 23.5 in the computed tables, a correction should be applied for the actual value, and in its absence accuracy can only be between the range of -13 to +24%.

D. G. HEWER.

Chemical sterilisation in the dairy industry. M. J. PRUCHA (World's Butter Rev., 1928, 2, 12—17).—The chlorine group is most satisfactory. The solutions employed were fairly stable up to 1 hr. at 26—71°; the addition of small amounts of milk caused a reduction in the amount of available chlorine. Addition of sodium hydroxide increased the stability in the absence, and decreased the stability in the presence, of milk.

CHEMICAL ABSTRACTS.

Manganese content of raw and cooked vegetables. S. MUNGER and W. H. PETERSON (J. Home Econ., 1928, 20, 194—200).—Spinach, string beans, and beets have a high, and rutabaga, cabbage, and onions a low, manganese content. The manganese content of stalks and leaves is generally higher than that of roots, tubers, and bulbs. The element is best conserved by steaming.

CHEMICAL ABSTRACTS.

Pectic constituents of tomatoes and their relation to the canned product. C. O. APPLEMAN and C. M. CONRAD (Md. Agric. Exp. Sta. Bull., 1928, No. 291, 1—17).—In green tomatoes protopectin predominates over pectin, but is rapidly transformed into pectin during the later ripening stages; from the pink to the red stage the ratio of pectin to protopectin increases from 1.13 to 2.56. Further conversion occurs by acid hydrolysis during processing and continues during cooling. Mid-season pickings, preferred for canning, showed the least transformation of protopectin to pectin.

CHEMICAL ABSTRACTS.

Biological values of certain types of sea foods. III. **Vitamins in clams.** D. B. JONES, E. M. NELSON, J. C. MURPHY [with J. P. DEVINE] (Ind. Eng. Chem., 1928, 20, 648—652).—Feeding experiments carried out on the same lines as those with oysters (B., 1928, 242) showed that clams are a good source of vitamin-A and -D, the soft-shell variety being somewhat richer in vitamin-A than the hard-shell variety. Compared with

oysters clams contain less vitamin-A but more of -D and -E. Unlike oysters they are practically devoid of vitamin-B.

F. R. ENNOS.

Digestibility of certain fruit by-products as determined for ruminants. S. W. MEAD and H. R. GUILBERT (Calif. Agric. Exp. Sta. Bull., 1927, No. 439, 1—11).

Digestibility and production coefficients of poultry feeds. G. S. FRAPS (Texas Agric. Exp. Sta. Bull., 1928, No. 372, 1—24.)

Water content of jelly. SOMIYA.—See VII.

PATENT.

Fertilisers from vegetable refuse (B.P. 291,514).—See XVI.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Tests for impurities in anaesthetic ether. E. B. ROBINSON (Lancet, 1928, 214, 856).—The aldehyde and peroxide tests first given by Wobbe (B., 1903, 1067) are described. The peroxide test is not as sensitive as that described by Middleton (B., 1924, 806), but is much simpler, and samples of anaesthetic ether which contain sufficient peroxide to respond to this test should be suspected.

E. H. SHARPLES.

Determination of atropine. E. RICHTER (Apoth.-Ztg., 1928, 43, 47; Chem. Zentr., 1928, i, 1076).—The determination of atropine in "granulæ atropine sulphur. 0.0005" is carried out by extraction of the aqueous solution with ether after addition of ammonia, evaporation of the ethereal extract, extraction of the atropine with absolute alcohol, dilution with water, and titration with 0.01N-hydrochloric acid, using methyl-red.

A. A. ELDRIDGE.

Assay of kalium sulphoguaiacolicum. E. RUPP (Apoth.-Ztg., 1928, 43, 74; Chem. Zentr., 1928, i, 1076—1077).—The procedure of the German Pharmacopœia VI is criticised, and modifications are proposed.

A. A. ELDRIDGE.

Evaluation of liquor kalii arsenicosi and the fixanal method. W. BÖRTGER (Apoth.-Ztg., 1927, 42, 1399—1400; Chem. Zentr., 1928, i, 1077).—Titrations with solutions of (a) iodine and potassium iodide, (b) iodine, potassium iodide, and a little hydrochloric acid, (c) iodate, iodide, and acid, gave concordant results. Discordant results reported with the fixanal method are ascribed to errors in its use.

A. A. ELDRIDGE.

Essential oils. SCHIMMEL & Co. (Ber. Schimmel, 1927, 4—5, 25—26, 53, 60—61, 66—67, 78, 85—86, 86, 114—115, 135; Chem. Zentr., 1927, II, 1518—1519).—**Araucaria oil.** The West Australian oil is a granular mass of a pleasant balsamic odour, having m.p. 30°, d_{25}^{15} 0.9764, $\alpha_D^{15} +14^\circ 25'$, n_D^{20} 1.50928, acid value 3.7, ester value 21.5, ester value after acetylation 173.6 (= 79.1% $C_{15}H_{26}O$), and is soluble in 3.5 vols. of 70% alcohol. From the fresh wood a yield of 10.5% of araucaria wood oil having d_{25}^{15} 0.9756, $\alpha_D^{15} +4^\circ 10'$, n_D^{20} 1.51139, acid value 10.2, ester value 24.3, ester value after acetylation 177.3 (= 81.1% $C_{15}H_{26}O$) is obtained. From the weathered wood 11.7% of oil having d_{25}^{15} 0.9742, $\alpha_D^{15} +8^\circ 5'$, n_D^{20} 1.51520, acid value 8.4, ester value 46.7, ester value after acetylation 186.7 (= 86.0% $C_{15}H_{26}O$).

$C_{15}H_{26}O$) is obtained. Both wood oils are soluble in 1.1 vols. of 80% alcohol. *Dill plant oil*. isoMyristicin (m.p. 44°) and myristicin have been identified in dill plant oil. By oxidation of isomyristicin with ozone, myristicin aldehyde (m.p. 131°) has been obtained. *Indian geranium oil*. A South Indian oil had d^{15}_D 0.9148, $\alpha_D - 8^\circ 31'$, n^{20}_D 1.47524, acid value 5.6, ester value 77.5 (32.6% of esters as geranyl tiglate), ester value after acetylation 210 (total geraniol 68.5%), solubility in 70% alcohol 1 in 2.5 vols. after rectification with steam the oil had d^{15}_D 0.9105 and n^{20}_D 1.47407. Oil from *Laurelia serrata* ("Huahuan"). The leaf oil of *L. serrata* grown in Chili had d^{15}_D 0.9469, $\alpha_D - 11^\circ 25'$, n^{20}_D 1.48731, acid value 0.8, ester value 8.4, solubility in 70% alcohol 1 in 2 vols. Oil from the bark had d^{15}_D 1.0426, $\alpha_D - 2^\circ 5'$, n^{20}_D 1.52482, acid value nil, ester value 6.5, solubility in 90% alcohol 1 in 0.5 vol. The characteristic constituent of both oils was safrol, the bark oil containing the most. *Mexican linaloe oil*. Steam-distillation of linaloe wood from Mexico gave 8.1% of oil having d^{15}_D 0.8781, $\alpha_D + 8^\circ 15'$, n^{20}_D 1.46072, acid value 1.5, ester value 47.5, solubility in 60% alcohol 1 in 4.5 vols., and consisting largely of *d*-linalool (75%). *Mexican petitgrain oil*. Leaf oil from sweet oranges had d^{15}_D 0.8675, $\alpha_D + 41^\circ 40'$, n^{20}_D 1.47407, acid value 2.4, ester value 12.1, and leaf oil from bitter oranges had d^{15}_D 0.8578, $\alpha_D + 42^\circ 55'$, n^{20}_D 1.47299, acid value 1.2, ester value 10.3. Both oils were soluble in 0.4 vol. of 90% alcohol, but not completely so in 10 vols. of 80% alcohol. The odour of both oils was stupefying and they did not appear to be suitable for use in perfumery. *Peppermint oil from Palestine*. Oil from the shade-dried plant had d^{15}_D 0.9096, $\alpha_D - 22^\circ$, n^{20}_D 1.46261, acid value 0.8, ester value 13.1 (menthol esters 3.6%), ester value after acetylation 154.9 (total menthol 48.8%), solubility in 70% alcohol 1 in 2.6 vols. (solution opalescent). Oil from sun-dried plant had d^{15}_D 0.9084, $\alpha_D - 21^\circ 17'$, n^{20}_D 1.46340, acid value 0.4, ester value 14.0, menthol esters 3.9%, ester value after acetylation 139.1, total menthol 43.3%, solubility in 70% alcohol 1 in 2.9 vols. (solution opalescent). Shade drying appears to be advantageous. *Pichurim bean oil*. From Brazilian Pichurim beans 2.1% of oil having d^{15}_D 1.0538, $\alpha_D - 3^\circ 30'$, n^{20}_D 1.52076, acid value 9.7, ester value 4.6, ester value after acetylation 44.8, solubility in 90% alcohol 1 in 0.5, was obtained. The oil had a saffras odour. Oil from *Geranium macrorrhizum*. The oil had m.p. $25-35^\circ$, d^{15}_D 0.9431—0.9638, $\alpha_D - 5^\circ 45'$ to $-7^\circ 38'$, n^{20}_D 1.50642—1.51538, acid value 1.2—1.5, ester value 5.6—14.9, ester value after acetylation 33.6—35.5, solubility in 80% alcohol 1 in 6—7 vols. (slight turbidity), in 90% alcohol 1 in 1 vol. The oil had a soft, crystalline consistency and after separation of the solid constituent (about 50%, m.p. $54-55^\circ$) had d^{15}_D 0.9460, $\alpha_D - 8^\circ 10'$, n^{20}_D 1.50698, ester value after acetylation 54.1. *Castoreum*. Steam-distillation of Canadian castoreum gave 2.1% of oil about half of which consisted of phenols. *o*-Cresol and a phenol, $C_{12}H_{16}O$, were detected, and, in the phenol-free oil, acetophenone, benzyl alcohol, and *l*-borneol. E. H. SHARPLES.

Adulteration of essential oils and natural perfumes. SCHIMMEL & Co. (Ber. Schimmel, 1927, 7, 8, 43—44, 48, 56, 64, 90, 93—94, 137—138, 144; Chem. Zentr., 1927, II, 1519).—The following adulterations

have been detected: birch-bud oil with methyl salicylate, cedar oil, and methyl phthalate; bitter almond oil with a crude benzaldehyde containing chlorine; lemon oil with 6% of spirits and 50% of lemon oil turpenes, and in another case with about 20% of mineral oil; a eucalyptus oil consisting almost completely of terpeneol first-runings; a cherry laurel oil consisting largely of benzaldehyde containing chlorine and containing no hydrocyanic acid; a lavender oil made from spike oil and methyl phthalate; rose oil with synthetic esters; East Indian sandalwood oil with phthalic and benzoic acid esters and with West Indian sandalwood oil, another sample consisting almost wholly of cedar oil, and another with a mineral oil fraction volatile in steam; juniper oil with nutmeg oil; liquid thymol consisting of oil of thyme and partly of carvacrol. E. H. SHARPLES.

Determination of citronellal in Java citronella oil. K. J. HOLTAPPEL (Perf. Ess. Oil Rec., 1928, 19, 210—212).—2 g. of oil in a 100 c.c. flask are cooled to about -2° and 20 c.c. of a neutral 5% alcoholic solution of hydroxylamine hydrochloride at a similar temperature are added together with bromothymol-blue. The flask is removed from the cooling bath and the contents are rapidly titrated with 0.5*N*-alcoholic potash. After the turning point 5 c.c. of alcoholic potash are added, the mixture is kept for 15 min. at ordinary temperature, and titrated with 0.5*N*-hydrochloric acid. Each c.c. of alcoholic potash corresponds to 77 mg. of citronellal. The results are, on the average, 1% below those obtained by Dupont and Rabaune's oximation method, which itself has a limit of error of at least 1%. The effect of refrigeration is to minimise the action of the liberated hydrochloric acid on the still unchanged citronellal, which action is accelerated by rise of temperature.

E. H. SHARPLES.

Determination of total geraniol in Java citronella oil. SCHIMMEL & Co. (Ber. Schimmel, 1927, 21; Chem. Zentr., 1927, II, 1519).—For the determination it is essential that acetic anhydride of higher percentage than 85% and completely dehydrated sodium carbonate be used and also that acetylation and saponification should each take at least 2 hrs. E. H. SHARPLES.

Extension of method for determination of essential oils in drugs. J. STAMM (Pharmacia, 1926, [5]; Chem. Zentr., 1927, II, 1519—1520).—Experiments have been carried out on the steam-distillation of known, varying amounts of essential oils with a constant quantity of water according to the author's method, and the quantity of essential oil which must be steam-distilled to give an oil yield in the oleometer similar to that of the oil yield from the corresponding drug has been established. From the results the actual amount of oil obtained from the drug under similar conditions can be corrected and the "true content" of essential oil determined. E. H. SHARPLES.

Determination of "true content" of essential oils in drug according to Stamm's method using the butyrometer. E. JÄGERHORN (Pharmacia, 1926, [5]; Chem. Zentr., 1927, II, 1520).—Gerber's butyrometer without grooves is used instead of Stamm's oleometer. The loss of carbon tetrachloride is an amount equal to that represented by one graduation

mark, and is added at the end. A 5 g. sample of the powdered drug is used. *Fructus foeniculi*, and *herba majoranae* and *caryophylli* gave larger yields of oil than those recorded in the literature; *fructus anisi* and *carvi* gave smaller yields. E. H. SHARPLES.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photogalvanic cell. IIMORI and TAKEBE.—See XI.

XXII.—EXPLOSIVES; MATCHES.

Nitration of glycerin. A. N. MASCHKIN (Ukraine Chem. J., 1928, 3 [Sci.], 59—113).—An examination of the results obtained by Nathan and Rintoul (J.S.C.I., 1908, 27, 193) leads to the conclusion that nitroglycerin is least soluble in an acid mixture of the composition $2\text{HNO}_3 + 5(\text{H}_2\text{SO}_4, \text{H}_2\text{O})$; if 92 pts. of pure glycerin are taken, the use of 841 pts. of a nitrating mixture containing 54.5% HNO_3 , 42.4% H_2SO_4 , and 3.4% H_2O should theoretically give a 227.6% yield. Better yields (i.e., 235.1%) should be given by reducing the quantity of acid to 558.6 pts. containing 44% HNO_3 and 55.9% H_2SO_4 , and still higher yields (236.2%) by taking 520.8 pts. of a mixture consisting of 43.4% HNO_3 and 56.6% H_2SO_4 . An acid mixture yielding a spent acid of low nitric acid content (5%) contains 41.2% HNO_3 and 58.6% H_2SO_4 , and gives a yield of 232.8% of nitroglycerin, the glycerin and nitrating mixture being taken in the proportions 1:5.4. Further confirmation of the theory that the best yields of nitroglycerin are obtained when the spent acid contains exclusively the monohydrate of sulphuric acid is afforded by the work of Soddy (Arms and Explosives, 1911, No. 222) and of Hofwimmer (B., 1911, 1333). A phase diagram constructed on the basis of results published by the above authors fully supports the contention that minimum solubility occurs along the line connecting pure nitric acid with sulphuric acid monohydrate.

R. TRUSZKOWSKI.

Determination of the stability of smokeless powder. M. TARLÉ (Bull. Chem. Soc. Japan, 1928, 3, 123—127).—Sources of error in the various methods used to determine the stability of smokeless powder are discussed. It is concluded that no satisfactory quantitative method is yet available.

C. J. SMITHELLS.

XXIII.—SANITATION; WATER PURIFICATION.

Availability of nitrate oxygen in filter effluents. O. M. URBAIN (Ind. Eng. Chem., 1928, 20, 634—635).—When sewage is diluted with aerated water containing nitrates, or, alternatively, a filter effluent containing nitrates is discharged into an aerated stream, the oxygen of the nitrate present is not available for purification purposes until the supply of dissolved oxygen is exhausted. The rate of consumption of oxygen from nitrates is slower than that of the oxygen in solution.

C. JEPSON.

Calcium-sodium ratio of certain [river] waters. R. A. HART (J. Amer. Ceram. Soc., 1928, 11, 314—316).—Under certain conditions, the removal of soluble materials from alkaline clay soils is rendered difficult

owing to the fact that the soil gradually becomes impermeable to water. This phenomenon is caused by the presence of sodium and potassium salts, and is prevented by calcium and magnesium salts in the soil or leaching water or in both. Clays deposited by water having a low calcium-sodium ratio have a greater density than similar clays deposited in water with a high ratio. A calcium-sodium ratio unfavourable to agriculture is favourable to ceramics. The tempering of clays with water having a low calcium-sodium ratio is suggested in order to produce a body of greater density.

F. SALT.

Interference of *Clostridium Welchii* with *B. coli* tests in water analysis. J. F. NORTON and M. BARNES (J. Amer. Water Works' Assoc., 1928, 19, 729—730).—Spores of *Clostridium Welchii* when present with *B. coli* in lactose broth cause a rapid production of acid and gas. When the acidity reaches p_H 4.2—4.3 in 24 hrs. the recovery of colon bacilli is uncertain, and with this acidity at the end of 48 hrs. these organisms are never viable. A possible interference in routine water examination is therefore indicated. C. JEPSON.

Comparative colon-aerogenes indices of water and sewage. R. E. NOBLE (J. Amer. Water Works' Assoc., 1928, 19, 733—746).—A comparative test of the cyanide-citrate pour-plate medium (cf. A., 1928, 318) and the standard lactose broth method involving 1051 samples of water of varying sanitary quality confirmed the opinion that the standard fermentation method was the less accurate. It was found that 76.7% of the samples examined by the plate method gave indices which equalled or exceeded those obtained by the broth method, 22.2% gave negative results with both tests, 13.4% were negative by the broth method and positive by the plate method, and 9.3% were negative by the plate method, but positive by the broth test. A further test in which a stored sample of polluted river water was used for the comparison on 37 successive days showed a steadier change in the *B. coli* content by the plate method than by the broth method.

C. JEPSON.

PATENTS.

Treatment of sewage and industrial waste. Purification of tannery waste. J. T. TRAVERS, Assr. to TRAVERS-LEWIS PROCESS CORP. (U.S.P. 1,672,584—7, 5.6.28. Appl., [A] 20.5.27, [B] 23.5.27, [C] 2.8.27, [D] 3.2.28).—Sewage is treated in (A) with dust from the precipitators used in cement manufacture; in (B) with solid waste from the manufacture of chlorine, chloride of lime, and caustic soda; and in (D) by adding a suitable electrolyte-producing material and increasing the hydroxyl-ion concentration so as to give all colloids a negative charge. In (C) tannery wastes from various parts of the plant are combined so as to neutralise each other as far as possible, and after removal of the suspended matter the liquor is treated with a reagent which will precipitate the colloids and liberate nascent oxygen to carry the purification process further. C. JEPSON.

Respirators. DEUTS. GASGLÜHLICHTAUER-GES. M.B.H. (B.P. 271,478, 17.5.27. Ger., 20.5.26).

Grading of materials (B.P. 290,422).—See I. Respirators (G.P. 447,425).—See VII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 17, 1928.

I.—GENERAL; PLANT; MACHINERY.

Calorimeter for determination of heats of combustion. A. EUCKEN and L. MEYER (Chem. Fabr., 1928, 177—179, 195—196).—The apparatus comprises a heavy hollow copper cylinder (250 c.c.) with copper lid, carrying the usual ignition apparatus terminating in a thin nickel wire. The material to be burnt (30—40 mg.) is mixed with 6—8 mg. of kieselguhr and pressed into a small briquette which is placed in a conical silver wire cage in the calorimeter and burnt in oxygen under atmospheric pressure. The temperature is recorded to the nearest one thousandth of a degree every $\frac{1}{2}$ min. until constant. A series of results showing the range of utility of the apparatus is given.

A. R. POWELL.

Apparatus for sampling powdered solids. J. VON MEERSCHIEDT-HÜLLESSEN (Chem. Fabr., 1928, 357—358).—A four-sided pyramid of sheet iron standing upright is provided with wings on each angle so as to direct material sliding down each face into a separate container. A funnel-shaped container above the pyramid is held centrally above its axis by a wooden staff which also closes the bottom of the funnel, and the whole is encased in a cylinder. The funnel is filled with the sample to be reduced, and the wooden plug lifted when the sample is automatically "quartered."

C. IRWIN.

Wetting [of powders] and its measurement. E. F. GRIEG (Fuel, 1928, 7, 136—138).—The "wetting" of a powder is measured by the rate of rise of the liquid up a column of the powder in a tube, the lower end of which just dips into the liquid. The tube is suspended from one arm of a modified Odén continuous-weighing balance, and its increase in weight determined at suitable intervals. The resistances of the column of powder to air flow and to liquid flow are also determined.

A. B. MANNING.

Device for the preparation of solutions of constant density. J. HAMOUS (Z. Zuckerind. Czechoslov., 1928, 52, 448).—An apparatus for diluting a liquid to constant density consists of a cylindrical vessel containing a float carrying a graduated spindle, which, according to its rise or fall, actuates a mechanism controlling the entrance of the diluting liquid at the point where the denser liquid is admitted.

J. P. OGILVIE.

Rapid evaporation at room temperature. E. JANTZEN and H. SCHMALFUSS (Chem. Fabr., 1928, 373—375, 390—392).—The principles involved in the construction of a high-capacity laboratory evaporator (40 litres of water per hr. at 15°) are discussed in detail. To attempt to remove water vapour by simple pump action is very uneconomical. It is better to use a

water-jet pump with a condenser. The connexion from the pump to the apparatus must be as short and wide as possible. A multitubular copper condenser is far more efficient than one of glass. The connexion from the evaporator to the condenser should be as wide as possible without any baffling arrangement to prevent splashing, which is guarded against by the low gas velocity and slight pressure drop. The still should be of simple construction of steel, copper, or aluminium. An apparatus of 30 litres still capacity constructed on these lines is described.

C. IRWIN.

Viscosity measurements and viscosimeters. S. ERK (Petroleum, 1928, 24, 830—834).—The viscosimeters of Couette, Engler, Vogel and Ossag, and Lawaczeck are described and illustrated.

W. S. NORRIS.

PATENTS.

Mixing or emulsifying apparatus. J. MCGOUGAN and J. HUNTER (U.S.P. 1,671,868, 29.5.28. Appl., 26.6.26. U.K., 4.7.25).—The apparatus comprises a chamber containing two interacting toothed rollers which serve as emulsifiers, and a feed hopper connected by means of a pipe to the emulsifying chamber. The discharge pipe from the latter is provided with an auxiliary branch by means of which the emulsion may be returned to the hopper and thence back to the chamber for re-treatment if desired.

A. R. POWELL.

Hardening of binder materials. [Manufacture of brake linings etc.] A. E. WHITE. From RAYBESTOS Co. (B.P. 292,371, 20.7.27).—Tape woven, e.g., from brass wire covered with asbestos is passed successively through a solution of a binding material, e.g., a 30% solution of gilsonite in gas oil, and the solvent is expelled by passing the impregnated tape through a heated liquid which does not dissolve or combine with the binder. The tape is cooled in an atmosphere of steam.

L. A. COLES.

Distillation of liquid mixtures. J. P. FISHER, ASST. to HEAT-TREATING Co. (U.S.P. 1,672,978, 12.6.28. Appl., 7.10.22).—The liquid passes downwards through a series of zones of increasing temperature, disposed one above the other. In order to facilitate the removal of vapours, the liquid is spread over an extended surface in each zone, and heat is applied to the liquid as it passes in a confined stream to the next zone below. The vapours evolved from each zone are condensed, and the heat of vaporisation is utilised to heat the liquid entering the cooler zone immediately above.

F. G. CLARKE.

Use of atmospheric pressure for regularly and continuously supplying liquid to [distillation]

vessels. L. GOURSAT and F. MICHAUD (F.P. 623,636, 25.10.26).—A closed supply container above the distillation vessel is connected with the latter by a pipe which just dips below the surface of the liquid. As the latter distils over, air enters the supply vessel by means of this pipe and delivers a corresponding volume of fresh liquid to the distillation vessel. A. R. POWELL.

Fractional precipitation of the products of distillation from gases. IURGI APPARATEBAU-GES.M.B.H. (G.P. 447,062, 17.9.24).—The hot gas mixture obtained from destructive distillation processes is fractionally cooled, and the resulting mist at every stage of the cooling is removed in an electrostatic precipitation apparatus, the purified gas from any one stage being enriched in constituents of lower b.p. by being passed back into an earlier stage of the process. A. R. POWELL.

Air separator. T. J. STURTEVANT, Assr. to STURTEVANT MILL Co. (U.S.P. 1,673,343, 12.6.28. Appl., 2.8.27).—Materials are graded in a separator comprising an inner and outer casing having a settling chamber between them. This chamber is situated in the inner casing, which consists of an upper and a lower shell separated by a series of spaced vanes. Material accumulating in the latter is removed pneumatically. F. G. CLARKE.

Crucible. E. L. HAUMAN (U.S.P. 1,673,115, 12.6.28. Appl., 13.11.26).—In the upper edge of the crucible is a semi-circular outlet, in line with which is arranged a trough-shaped lip, the bottom being slightly below the lower part of the outlet. H. ROYAL-DAWSON.

Hot filter funnel. P. SEGEBADE (G.P. 447,136, 19.4.25).—A Büchner funnel for hot filtration is totally enclosed within a container of similar shape through which heating liquid is passed. A. R. POWELL.

Protection of water pipes. C. BÜCHER (B.P. 265,932, 10.1.27. Ger., 12.2.26).—Suitable apparatus for carrying-out the process of B.P. 260,233 (B., 1927, 240) is described. F. G. CROSSE.

Reversible regenerative furnace. J. S. ATKINSON, Assr. to OPEN HEARTH COMBUSTION Co. (U.S.P. 1,675,588, 3.7.28. Appl., 8.8.21. U.K., 22.9.13).—See B.P. 22,678 of 1913; B., 1915, 875.

Gas liquefiers for refrigerating plants. SILLER & RODENKIRCHEN G.M.B.H., and W. SILLER (B.P. 292,402, 15.10.27).

Optical pyrometer (B.P. 291,971). Corrosion of metallic parts (U.S.P. 1,671,173).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Replaceable bases in the roofs of bituminous coal seams of Cretaceous age. E. McK. TAYLOR (Fuel, 1928, 7, 127—128; cf. B., 1928, 288).—Examination of the roofs of such bituminous coal seams shows them to have undergone base exchange with solutions of sodium salts, the sodium aluminosilicic complex having subsequently been hydrolysed in fresh water. A. B. MANNING.

Replaceable bases in the roofs of bituminous coal seams of Tertiary age. E. McK. TAYLOR (Fuel, 1928, 7, 129—130; cf. preceding abstract).—A few

specimens of the roofs of bituminous coal seams of Tertiary age have been examined. They exhibit characteristics similar to those shown by the roofs of other bituminous coal seams. A. B. MANNING.

Conditions and geological evidence of base exchange between the roofs of bituminous coal seams and solutions of sodium chloride. E. McK. TAYLOR (Fuel, 1928, 7, 130—134; cf. preceding abstracts).—From considerations based on the type of fossils found in the roofs of coal seams it is concluded that base exchange may have taken place in three ways: (a) by deposition of the roof in salt water, (b) by the submergence of a fresh-water roof in the sea, and (c) by capillary action from subsoil water containing sodium chloride. If it is accepted that, although the modes of accumulation of the plant residues may have been widely different, the roof conditions have been the final factor in their transformation into coal, it becomes possible to reconcile the opposing theories of the "drift" and "*in situ*" origin of coal. A. B. MANNING.

Brown coals of the Cheliaba region. G. L. STADNIKOV and N. F. PROSKURNINA (Trans. Karpov Inst. Chem., 1926, No. 5, 95—99).—Cheliaba coals, although externally similar to typical coals, are classed as brown coals; they contain C 71.51—76.05, H 4.40—4.97, N 1.32—2.00, S 0.84—3.53, O 16.49—20.61%, and yield moisture 16.9—19.0, ash 7.3—23.4, volatile matter 44.1—52.3, coke 47.7—55.9, S 0.75—2.52, resins 0.8—3.2%. CHEMICAL ABSTRACTS.

Resins of Wealden lignite. R. WIGGINTON (Fuel, 1928, 7, 118; cf. B., 1927, 3).—The ether-soluble resin of a sample of Wealden lignite, amounting to 1.5% of the original material, has been fractionated by extraction of the ethereal solution with dilute solutions of ammonium carbonate, sodium carbonate, and potassium hydroxide, in succession, followed by precipitation of part of the neutral resin by the addition of light petroleum. Each fraction has been analysed. A silver salt, prepared by the addition of silver nitrate to a neutral solution of the potassium salt of one of the resin acids, has also been analysed. A. B. MANNING.

Thermal insulation of retort settings. A. J. DALE and A. T. GREEN (Gas J., 1928, 183, 37—42).—Different methods of improving the thermal insulation of retort settings are critically discussed. Regarding the insulation of horizontal settings the guiding principles of design should be: (a) to use a diatomite type of brick, (b) to include wall insulation within the wall, away from external disruptive influences and sufficiently distant from the inner face to prevent undue shrinkage and loss of insulating power, (c) to ensure a rigid tying of the inner and outer firebrick walls, (d) to guard against overheating of metal fittings, mouthpieces, etc., (e) to use insulating plugs in the sight holes, and (f) to apply for top insulation a 4½-in. course, on the outside of the roof for horizontal settings, but preferably one course below the top course for vertical-retort settings. Tongued-and-grooved shapes should be used, so that if the joints open, direct radiation from the hot face is obviated. When the design involves external insulation it is advisable to build in the insulating brickwork after the

setting has attained working temperatures. A considerable decrease in the thermal efficiencies of plants fitted with waste-heat boilers or external producers can occur unless the gas mains also are efficiently lagged.

A. B. MANNING.

Relation between low- and high-temperature carbonisation of coal. A. SHIMOMURA (Fuel, 1928, 7, 119—127).—Seven Japanese coals of different types have been carbonised in the laboratory at 500°, and the semi-cokes then further carbonised at 900°. The volatile matter content of a coal is of little value for determining its behaviour on carbonisation. Water of constitution is driven off during both stages of the carbonisation, coals of lower rank yielding more water during the first than during the second stage, whilst the reverse is true of high-grade bituminous coals. All the tar is driven off below 500°. The yield of ammonia in the first stage is only 10—25% of that in the second, the total yield representing about 20% of the total nitrogen. The volume of gas evolved from 500° to 900° is 2—2.5 times that evolved up to 500°; the former consists largely of methane and hydrogen, contains more carbon monoxide than dioxide, and no unsaturated hydrocarbons.

A. B. MANNING.

Geipert's method for the determination of the gas yield in therms from gas coals. K. BUNTE and W. ZWIEG (Gas- u. Wasserfach, 1928, 71, 629—631; cf. B., 1926, 938).—The method yields concordant results in the hands of different investigators using either the same or different sets of apparatus. The results agree closely with those of gas-works' practice. Some of the experimental details which require careful control in order to ensure concordant results are discussed.

A. B. MANNING.

Thomas recording gas calorimeter (Dept. Sci. Ind. Res., Fuel Res., Tech. Paper No. 20, 1928, 42 pp.).—The results of experiments of a fundamental nature carried out on the earlier models indicate that (1) with gas of constant calorific value the record made by the calorimeter is correct within about 1%; (2) when the calorific value alters, there is a lag of about 30 min. before the change appears to its full extent on the record, small or temporary changes being ignored or under-estimated; over long periods the average of the record agrees satisfactorily with values obtained on a Simmance machine; (3) variations of gas pressure, voltage of electric supply, room temperature, and barometric pressure, within reasonable limits, do not upset the performance. After these tests the instrument was altered in the heat-absorbing air meter, where disturbance of the water level was lessened by changes in the construction of the vanes and of inlet and outlet ports, in the constant-level water feed where incoming water was made to splash on to the overflow weir, and in the burner which was constructed of silica instead of metal. Finally, changes were made in the recording mechanism by which it became more sensitive and less liable to lag and to irregular working. Tests on the modified apparatus indicate that (1) a steady reading can be obtained about 20 min. after lighting the burner, and if the calorific value of the gas is changed this will appear within about 4 min., but only to its full extent after

about 15—20 min.; at more frequent intervals than this changes are not recorded to their proper value, although the periods and the average will be correct; (2) fluctuations in the speed of the driving motor have small permanent disturbing effects, but may cause larger temporary errors; (3) changes in the level of the water in the tank must be avoided as far as possible, as a decrease of even 0.1 in. produces a rise of about 18 B.Th.U. on the chart; (4) the recording mechanism is satisfactory, and no troubles were encountered in other mechanical parts; the instrument is more accurate than water-flow or differential-expansion types, although its time lag is greater.

R. H. GRIFFITH.

Determination of dust and ash [fixed carbon] in tar. R. ARBEITER (Chem.-Ztg., 1928, 52, 529).—The tar sample is placed on a thick filter paper supported by a porcelain crucible (with no bottom) which rests on a glass tripod. The whole is placed in a large beaker containing carbon tetrachloride, and having resting on it a round-bottomed flask through which cold water flows. Heating is by an electric hot plate, and extraction is complete in 30—40 min. The filter is then dried, weighed, and, if desired, burnt.

C. IRWIN.

Liquid-phase cracking [of hydrocarbons]. A. DOBRJANSKI and A. MUREJEVA (Neftjanoe Chozjajstvo, 1927, 12, 411—413; Chem. Zentr., 1927, II, 1524).—Machine oil, cylinder oil, vaporising oil, and paraffin have been cracked in the liquid phase at 405—415°/atm. As the decomposition progresses, the quantity of ethylene and of heavier hydrocarbons decreases and that of saturated hydrocarbons increases. The density of the liquid distillate progressively increases, whilst the content of hydrocarbons of b.p. up to 150° decreases. Possibly during the earlier stages of decomposition the longest side-chains of the cyclic hydrocarbons are split off, this being followed by a different decomposition leading to the formation of saturated hydrocarbons. Pumice, coke, copper filings, and iron filings have no effect on the process of cracking.

W. S. NORRIS.

Cracking [of mazout] under low pressure. K. KOSTRIN (Neftjanoe Chozjajstvo, 1927, 12, 39—45; Chem. Zentr., 1927, II, 1523—1524).—Paraffin base mazout from Surachany has been cracked at 480—490° and 2.5—3.0 atm. in a tube furnace, which also served for the subsequent distillation, during which 33.5% of distillate was collected. The residue, 59.2%, had a setting point 12° lower than that of the original mazout, and was suitable for use as fuel oil. The distillate had initial b.p. 90°, and 39% was volatile up to 150°. The gasoline obtained therefrom contained 13% of unsaturated hydrocarbons; this value was halved and the boiling range raised by treatment with Florida earth, which polymerised the unsaturated constituents. Sulphuric acid produced a similar but even greater effect, and could not be used as a refining agent.

W. S. NORRIS.

Refining of mineral oils. F. SCHWARZ (Petroleum, 1928, 24, 803—810).—In place of solutions of alkali naphthenates, which are capable of dissolving easily emulsifiable and/or gum-forming hydrocarbon constituents of light lubricating oils, the author makes use of solid soaps upon which gum-forming unsaturated

constituents are adsorbed (cf. G.P. 403,134—5; B., 1925, 64). The use of this reagent may supplant or supplement refining by means of sulphuric acid, or it may replace the usual treatment (following acid) with alkali hydroxide solution or with dry adsorbents. Treatment with precipitated soaps, if used in conjunction with acid refining, preferably follows the latter, although the soap treatment can be given first. W. S. NORRIS.

Manufacture of "contact substance" [from petroleum distillates]. B. VLASSENKO (Neftjanoe Chozjajstvo, 1927, 12, 542—549; Chem. Zentr., 1927, II, 1525).—A report dealing with the large-scale preparation of "contact substance," consisting of about 40% of naphthenesulphonic acids, 2% of sulphuric acid, and 15% of oil and water, obtained as a by-product in the refining of petroleum distillates with fuming sulphuric acid. W. S. NORRIS.

Transformer oils and their brown deposit. G. L. STADNIKOV and Z. I. VOZZINSKAJA (Trans. Karpov Inst. Chem., 1926, No. 5, 109—126).—The brown deposit in used transformer oils is formed by condensation of the resinous with organic compounds formed by oxidation of the oil. Sulphonic acids remaining in insufficiently washed refined oils catalyse the condensation. The behaviour on oxidation of oils treated with 90% and fuming sulphuric acid in the presence or absence of naphthalenesulphonic acids supported this view.

CHEMICAL ABSTRACTS.

Valenta's reaction in relation to its use with gasolines and benzene-benzene mixtures. R. WELSLER (Petroleum, 1928, 24, 768—773).—The solubility of benzene, amylene, cyclohexane, and octane in 1.5 vols. of methyl sulphate at 20°, respectively, is 100, 72, 13.5, and 5.5%. The soluble (9%) and insoluble portions of Kahlbaum's "normalbenzin" are practically identical with the original material. Between —20° and +60° the variation in solubility of a commercial gasoline is linear with the temperature. Experiments are recorded which show that treatment of methyl sulphate under a variety of conditions will effect only a partial separation into soluble and insoluble portions. Methyl sulphate is therefore useless as a means of investigating mixtures, or of determining the content of aromatic hydrocarbons or other anti-knock constituents of a commercial gasoline. W. S. NORRIS.

Viscosity-gravity constant of petroleum lubricating oils. J. B. HILL and H. B. COATS (Ind. Eng. Chem., 1928, 20, 641—644).—From data previously published (cf. Hill and Ferris; B., 1926, 84) on the physical properties of the viscous fractions from various types of crude petroleum, a mathematical relation has been worked out between Saybolt viscosity and sp. gr. which gives a constant which has a calculable value for any oil and is an index of its chemical composition. As the value is higher or lower so the character of the oil becomes more naphthenic or more paraffinic, respectively. Furthermore, the higher the constant, the greater is the viscosity change with temperature. The relations hold for distillates of fairly narrow boiling range and for blended oils from components of widely different viscosities, but fractions from a mixture of several crudes may give abnormal results.

H. S. GARLICK.

Apparatus for determining the setting point of mineral oils. ANON. (Chem.-Ztg., 1928, 52, 529).—A Dewar flask containing ether is fitted with a tube in which the oil sample is placed and having the necessary thermometers and air connexions. The inlet air is dried with calcium chloride and connected to a reservoir of ether, and the outlet to a filter pump. The temperature difference between the ether and oil should not exceed 5°. Temperatures are easily controlled, and can be obtained as low as —40°. C. IRWIN.

Oxidation of "vaseline" oil. G. S. PETROV, A. J. DANILOVICH, and A. Y. RABINOVICH (Trans. Karpov Inst. Chem., 1926, No. 5, 81—89).—Purified (sulphuric acid) "vaseline" oil, when heated and refined with alkalis, is not oxidised when exposed to a current of air for 49 hrs. at 100° in presence or absence of zinc or iron. In presence of lead, oxy-acids insoluble in benzene are formed. Commercial "vaseline," when heated for 10 hrs. at 250—270°, shows signs of oxidation, but distilled oils (although not purified with sulphuric acid) do not. Oxidation of the oils, yielding acids soluble in benzene, is best effected below 100° in presence of cobalt naphthenate. CHEMICAL ABSTRACTS.

Determination of ceresin in ozokerite [mineral wax] and paraffin goudron. W. TOKMANOV (Neftjanoe Chozjajstvo, 1927, 12, 558—561; Chem. Zentr., 1927, II, 1525).—The ozokerite, dissolved in benzene, is treated for 1.5 hrs. at 60—65° with sulphosil (B., 1928, 602); the portion not absorbed is ceresin. This procedure gives higher values for ceresin, when applied to crude ozokerite, than the method of Lach ("Die Ceresinfabrikation," 1911, p. 60), but gives somewhat lower figures when applied to ozokerite previously freed from liquid constituents. In Lach's method, which entails heating at 200°, volatile hydrocarbons are lost which are not absorbed by the sulphosil; on the other hand, the separation of resin from ozokerite by sulphosil is more complete. Silica gel or Florida earth adsorbs resin already present, but, unlike sulphosil, does not polymerise and completely remove unsaturated, resin-forming constituents. If ozokerite is treated first with silica gel and then with sulphosil, separation into resin, resinifying constituents, and ceresin is effected.

W. S. NORRIS.

Solid-solution formation in mixtures of paraffin waxes. L. D. MEYERS and G. STEGEMAN (Ind. Eng. Chem., 1928, 20, 638—641).—Equilibria existing between mixtures of waxes separated from one another by a series of fractional distillations were determined from the temperatures at which crystals first appeared when melts were slowly cooled. The crystals varied in composition according to the original mixture from which they separated—a characteristic of solid solutions. The purest wax obtainable possessed no definite m.p. Results suggest that wax mixtures form a complete series of solid solutions the freezing points of which lie between those of the components. Intersolubility of wax and oil is low up to about 10° below the m.p. of the wax. Increased solubility at high temperatures accounts for the great loss of wax in the later stages of the sweating process. Purification of crystals separating from fused wax-oil mixtures by a series of pressings was resorted to. Since a wax containing 2.5% of oil produced an oil spot on

filter paper, whilst a wax to which 2% of oil had been added could be subjected to 3000 lb./in.² pressure without doing so, it is concluded that the oil is retained in the form of a solid solution. H. S. GARLICK.

Calorimeter. EUCKEN and MEYER.—See I. **Activated silica gel.** TYTSCHININ and TOKMANOV.—See VII. **Preservation of wood.** VORONOV.—See IX.

PATENTS.

Treatment of coals, ores, etc. mainly composed of particles of small sizes. L. HOYOIS (B.P. 279,447, 18.10.27. Belg., 19.10.26).—The material is separated into sharply distinct grades by an apparatus comprising trough washers and a specially designed upward-current washer, the general principle of the method of separation being first to divide the material into two portions of less and greater density, respectively, than the average, and then to subdivide these, separating from the former all particles of density greater than the permissible limit for the light particles, and from the latter all particles of density less than the permissible limit for the heavy particles. These two portions are then combined to form a middle portion. A. B. MANNING.

Production of stable suspensions or pastes of coal. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 292,217, 11.3.27).—The finely-ground coal is mechanically dispersed in an alcohol, *e.g.*, crude butyl alcohol, with the addition of a basic substance (sodium hydroxide, pyridine, aniline) as stabiliser. The stability of the suspension may be further improved by the addition of a small quantity of mineral oil.

A. B. MANNING.

Preparation of solid fuels for burning in pulverised form. L. B. GREEN, Assr. to BORDEN CO. (U.S.P. 1,673,114, 12.6.28. Appl., 20.11.26).—The fuel is pulverised, then briquetted, and the surfaces are freed from loose fuel before delivery to a burner.

H. ROYAL-DAWSON.

Production of briquettes. W. T. MILLER, Assr. to U.S. COAL MANUF. CO. (U.S.P. 1,670,865, 22.5.28. Appl., 10.10.23).—Finely-divided carbonaceous material is mixed with a binding and waterproofing composition, consisting of a mixed solution of a soluble silicate and glucose or dextrin, the latter ingredient having been partially precipitated by means of alcohol. Sodium chloride or other volatilisable salt may also be present. The plastic mass is moulded under pressure and baked.

F. G. CLARKE.

Manufacture of fuel briquettes. II. H. HANSARD U.S.P. 1,675,266, 26.6.28. Appl., 23.6.24).—A mixture of pulverised coal and a liquid hydrocarbon is heated to remove the constituents of low b.p. of the latter, and the resulting mass is briquetted. H. ROYAL-DAWSON.

Composition fuel [briquettes]. H. V. T. MILLS and V. D'O. NOBLE (B.P. 291,958, 8.8.27).—A mixture of small coal, cement, sawdust, and creosote is used.

F. G. CROSSE.

Active carbon. SOC. ANON. DES ENGRAIS ET NOIR ANIMAL (F.P. 623,455, 18.2.26).—Carbonised wood turnings are heated at 300–500° with a mixture of sulphuric and phosphoric acids. A. R. POWELL.

Active carbon. GES. F. CHEM. PROD. M.B.H. (Austr. P. 106,603, 29.9.22).—In the preparation of alkali sulphides from alkali sulphates a large excess of organic reducing substance is employed in the fusion, and the carbon resulting therefrom is obtained as a sludge after leaching out the soluble salts from the fused sulphide mass.

A. R. POWELL.

Manufacture of activated charcoal or carbon. METALLBANK & METALLURGISCHE GES. A.-G., and E. SCHELLER (B.P. 292,039, 4.1.28).—Wood, peat, sugar, inactive charcoal, or other carbonaceous material is heated with sodium monoxide. The initial reaction proceeds automatically after being started by local heating; when the reaction has subsided the product is heated at 500–700°. The sodium monoxide may be used alone or in conjunction with other substances such as sodium chloride, hydroxide, or carbonate, the salts remaining at the end of the reaction being removed by washing.

A. B. MANNING.

Distillation of coal and other solid carbonaceous materials. T. A. GOSKAR (B.P. 291,836, 1.3.27 and 29.6.27).—Bituminous coal (20–40 pts.) and a binding agent (8–15 pts.) consisting of a solid hydrocarbon of the paraffin or terpene series, *e.g.*, bitumen or resin, are reduced to a fine state of division and intimately mixed. Finely-ground coal waste, anthracite duff, or coke breeze, etc. (70–50 pts.) is then added, the mixture briquetted, and distilled in a retort internally heated by the passage of hot producer- or water-gas, with or without superheated steam.

A. B. MANNING.

Distillation of solid carbonaceous materials. H. NIELSEN and B. LAING (B.P. 292,060, 4.12.26. Cf. B.P. 276,407, 287,037, and 287,381; B., 1927, 867; 1928, 356, 395).—Coal is subjected to the action of hot gases containing 2–8% of oxygen in order to modify its coking properties, and is then distilled in a current of an inert, gaseous, heating medium. The distillation may be carried out in two stages, first at a temperature not exceeding 600° and then at a high temperature (up to 1200°). The solid residue is finally gasified in the presence of steam.

A. B. MANNING.

Distillation of bituminous substances, fuels, etc. R. HADDAN. From KOHLENVEREDLUNG A.-G. (B.P. 291,481, 20.1.27).—The finely-divided raw material is maintained in suspension in a stream of heating gas which is either itself a hydrogenating agent or into which hydrogen or other hydrogenating agent is introduced. In one method of working, steam is added to the heating gas, the temperature being such that the fuel interacts with the steam to produce water-gas. Pressures higher than atmospheric may be used to aid hydrogenation.

A. B. MANNING.

Distillation of solid fuels. C. STILL (G.P. 446,173, 14.5.25).—The material is heated in long vertical retorts, and the volatile products formed are removed from the lowest part of the retort. The retort is heated in such a way that, in the region of the outlet pipe for the gases, a premature progress of the coking from the walls towards the centre is avoided.

A. R. POWELL.

Retort for the distillation of bituminous solid fuels, in particular bituminous shale, coal, peat,

etc. G. MENELL (B.P. 291,550, 17.3.27).—A horizontal rotary retort is fitted throughout its length with a spiral web, made of suitably bent semi-circular sheet-iron vanes bolted or riveted together. Longitudinal plates are fitted between the spiral threads of the web, extending from the inner surface of the retort towards the centre. The retort is particularly adapted for the treatment of shale which has a tendency to cake.

A. B. MANNING.

Carbonisation of coal. S. DUNLOP. From M. J. McQUADE (B.P. 291,962, 14.6.27).—Carbonisation is carried out at 350—450° in a horizontal cylindrical retort along which the coal is alternately advanced a certain distance and retracted a shorter distance by means of a screw conveyor. A space is left above the coal, and the volatile products which collect therein are drawn off through suitably placed offtake pipes. A. B. MANNING.

Treatment of gases arising from the distillation of coal etc. C. COOPER (B.P. 291,832, 11.2.27).—The gases are first submitted to a process for reducing their moisture content (cf. B.P. 248,841; B., 1926, 428) and are then oil scrubbed for the removal of naphthalene. The degree of dehydration may be merely sufficient to secure the maximum efficiency of the subsequent oil scrubbing, or, if desired, may be carried a stage further in order to prevent condensation of water in the distributing mains.

A. B. MANNING.

Apparatus for enriching with oil the gas obtained by the complete gasification of carbonaceous fuel. REGENERATIVE COAL GASIFICATION SYSTEM, LTD., and M. W. TRAVERS (B.P. 292,410, 11.11.27).—The apparatus described in B.P. 210,356 (B., 1924, 244) is modified by placing the regenerating and carburetting chambers in series and passing the whole of the blow gas through them in succession. If desired, the carburettor may be divided into two chambers, the carburettor proper being followed by a superheater.

A. B. MANNING.

Gas generators. H. S. and W. S. MOORE (B.P. 292,408, 10.11.27).—A plant for the production of coal gas, water-gas, mixed gas, carburetted water-gas, or low-grade gas, according to the method of operation, consists of a vertical retort below which and in open communication with which is a water-gas generating chamber. The fuel is either partially or totally gasified; in the former case the coke is discharged through a semi-rotary extractor into a chamber in which it is quenched. The retort is heated by the blow gases which are burnt with secondary air and then pass through a series of annular superposed chequered recuperative heating chambers surrounding the retort. The water-gas produced during steaming passes up through the retort, aiding the carbonisation process and mixing intimately with the coal gas evolved. One or more of the recuperative heating chambers can be utilised as carburetting chambers if it is desired to enrich the gas.

A. B. MANNING.

Production of labile bitumen emulsions. RÜTGERSWERKE A.-G., and L. KAHL (B.P. 275,928, 18.3.27. Ger., 13.8.26).—Emulsions for tarring roads are produced by mixing a basic magnesium salt to a paste with water and then stirring into this the liquid tar or asphalt.

Such an emulsion can be diluted with water and applied to the road surface irrespective of temperature or weather.

A. B. MANNING.

Treatment of bitumen and other materials adaptable for insulating or dielectric purposes. D. ANDERSON & SON, LTD., and R. O. CHILD (B.P. 291,858, 16.8.27).—In order to free bitumen, tar, etc. from suspended matter and thereby improve its insulating properties, it is treated with a colloid, e.g., casein, in a partially coagulated state, and the mixture centrifuged. If necessary the material is heated during the process.

A. B. MANNING.

Manufacture of asphalt. E. A. RUDIGIER, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,673,533, 12.6.28. Appl., 19.8.22).—Hot petroleum residuum at about 216° is transferred to a heat-insulated chamber and air-blown without further application of heat.

C. O. HARVEY.

Decolorisation of acetone oils and wood spirit distillates. ÉTABL. LAMBIOTTE FRÈRES (F.P. 619,857, 14.12.25).—The coloured oils are heated under reflux with a metal chloride (chloride of calcium, magnesium, zinc, aluminium, iron, tin, or antimony) at ordinary or increased pressure; the coloured constituents are converted into resinous or tarry products, and the oil is obtained colourless by distillation. Examples are: heavy acetone oil (b.p. 125—200°) with anhydrous magnesium chloride, and a wood spirit fraction (b.p. 125—195°) with anhydrous ferric chloride. C. HOLLINS.

Catalytic cracking of heavy hydrocarbons, mineral oils, oil residue tars, and the like. E. ERLÉNBAUGH, Assr. to SINCLAIR REFINING CO. (U.S.P. 1,671,573, 29.5.28. Appl., 15.8.22. Ger., 22.9.21).—The formation of coke during catalytic cracking is prevented by circulating the heated oil repeatedly through a deep layer of the catalyst resting on a perforated support within the still. The vapours pass off through a dephlegmator, and a viscous residue is removed from the still.

C. HOLLINS.

Cracking of mineral crude oils. H. BLUMENBERG, JUN., Assr. to A. M. BULEY (U.S.P. 1,673,491, 12.6.28. Appl., 18.5.27).—Aluminium chlorosulphate is added to the oil, the mixture is heated to cracking temperature and distilled, and the distillate condensed.

H. ROYAL-DAWSON.

Production of olefines. A. W. NASH, A. R. BOWEN, and O. C. ELVINS (B.P. 291,867, 9.3.27).—Mixtures consisting substantially of olefines and paraffins are produced from gases containing carbon monoxide and hydrogen, in which the proportion by vol. of the former exceeds that of the latter, by passage at atmospheric pressure and at 280—300° over a granular catalyst consisting of a reduced mixture of the oxides of cobalt, copper, and manganese. The catalyst is prepared by precipitation of the oxides or ignition of the nitrates etc., followed by reduction with hydrogen at 400—420°, and may be revived after use by treatment with steam or by oxidation and subsequent reduction. E.g., a suitable catalyst is produced by the reduction of a mixture containing copper oxide, cobalt oxide, and manganese oxide in the proportions by wt.: 30:60:45. If the gases are in equal proportion (ordinary water-gas), or if

the hydrogen is in excess, the products are mainly paraffins. C. O. HARVEY.

Production of nitrogenous-base oils from hydrocarbon materials. H. K. IHRIG, Assr. to S. E. CAMPBELL and ASSOCIATED OIL CO. (U.S.P. 1,671,721, 29.5.28. Appl., 8.11.26).—The sulphur dioxide extract of mineral oil (especially Californian petroleum) contains most of the nitrogenous bases (pyridine, piperidine, quinoline) originally present. These bases are removed with dilute sulphuric acid and recovered. C. HOLLINS.

Separation of hydrocarbons. C. B. WATSON, Assr. to PURE OIL CO. (U.S.P. 1,673,854, 19.6.28. Appl., 26.12.25).—Cracking residues are forced by means of an inert gas through a conduit and atomised in an enlarged chamber, into which passes a further quantity of heated inert gas to maintain the temperature and promote turbulence in the material. The vapours are withdrawn and the unvaporised residue is recirculated, a portion of it being continuously consumed by a number of burners. C. O. HARVEY.

Refining of hydrocarbon oils. P. McMICHAEL, Assr. to HYDROCARBON REFINING PROCESS CO. (U.S.P. 1,669,944, 15.5.28. Appl., 12.1.25).—Petroleum oils and distillates are agitated with 10% of their volume of a solution of sodium hydroxide (*d* 1.1) saturated with sodium hyposulphite, then with 10% of their volume of a 10% solution of sodium chromate containing 1% of sodium hydroxide, and finally with a 1% solution of hydrochloric acid. Instead of the first-named solution the liquid obtained by treating with lime the solution produced by reducing sodium hydrogen sulphite with zinc dust may be used. A. R. POWELL.

Purification of mineral and naphtha oils. G. PETROFF (B.P. 291,823, 1.2.27).—In the production of light-coloured odourless oils, such as transformer or turbine oils, by treatment with sulphonating agents, more complete removal of unsaturated and resinous substances and greater ease of separation of the sulphonic acids from the diluted acid tars and of the alkaline liquor during subsequent alkali treatment are attained by mixing fatty acids, *e.g.*, oleic or stearic acid, with the oil to be refined. C. O. HARVEY.

Manufacture of fuel briquettes [from peat]. L. RUDEMAN (U.S.P. 1,674,179, 19.6.28. Appl., 30.4.27).—See B.P. 236,366; B., 1925, 699.

Dewatering peat. G. A. SCHROTER (B.P. 292,661, 18.3.27).—See U.S.P. 1,625,058; B., 1927, 577.

Absorbent catalyst. A. GODEL, Assr. to SOC. DE RECHERCHES ET D'EXPLOIT. PÉTROLIFÈRES (U.S.P. 1,674,897, 26.6.28. Appl., 8.12.25. Fr., 9.12.24).—See B.P. 244,461; B., 1927, 133.

Production of carbon electrodes. I. SZARVASY (U.S.P. 1,675,674, 3.7.28. Appl., 16.3.27. Ger., 15.11.26).—See B.P. 284,818; B., 1928, 252.

Manufacture of gas from coal. M. W. TRAVERS and F. W. CLARK, Assrs. to TRAVERS & CLARK, LTD. (U.S.P. 1,674,000, 19.6.28. Appl., 5.3.23. U.K., 9.3.22).—See B.P. 198,777; B., 1923, 759 A.

Treatment of oils [hydrocarbons] with liquid sulphur dioxide. W. HESS, Assr. to ALLGEM. GES. F.

CHEM. IND. M.B.H. (U.S.P. 1,674,676, 26.6.28. Appl., 28.10.26. Ger., 29.3.26).—See B.P. 268,726; B., 1928, 181.

Separating from fluid hydrocarbons other hydrocarbons which precipitate at low temperature. N. O. BACKLUND, Assr. to BERGEDORFER EISENWERK A.-G. (U.S.P. 1,676,069, 3.7.28. Appl., 28.1.27. Ger., 16.8.26).—See B.P. 267,038; B., 1927, 357.

Separating the neutral oils in tars, tar oils, and pitches from acid constituents. H. WITTEK (U.S.P. 1,674,710, 26.6.28. Appl., 14.4.27. Ger., 12.8.25).—See B.P. 256,933; B., 1927, 901.

Pulverised fuel burners. H. CAMBERS (B.P. 291,842, 3.3.27).

Apparatus for generation of acetylene gas. J. HAWORTH (B.P. 291,872, 10.3.27).

Smoke-treating apparatus. C. J. SCHOBRONE (B.P. 292,380, 15.8.27).

Apparatus for generation of acetylene gas under low or high pressure. J. HAWORTH (B.P. 291,897, 17.3.27).

Gaseous mixtures (B.P. 281,675).—See VII. **Waterproof cement** (B.P. 291,988).—See IX. **Purification of mineral oils** (B.P. 291,817).—See XII.

III.—ORGANIC INTERMEDIATES.

Absolute alcohol in industry. J. L. GENDRE (Chim. et Ind., 1928, 19, 396—401; cf. B., 1925, 185, 563, 646).—Processes depending on the use of lime for the rectification of alcohol have now been abandoned, the method being retained only in the treatment of fermented bisulphite lyes in some paper factories. Even in this case losses of alcohol are excessive and there is risk of explosion. Lorette's process, using alcohol vapour and powdered lime, gives a turbid alcohol which must be freed from lime. Dehydration with a glycerol solution of potassium carbonate has been tried at Montières-les-Amiens, but the process requires the regeneration of anhydrous or nearly anhydrous glycerol—an operation involving the use of vacuum and live steam at 145—150°. Processes depending on the distillation of azeotropic mixtures are discussed and compared. Comparative costings show that rectification is practically as costly a process as the manufacture of absolute alcohol from phlegms, and, accordingly, methods based on rectification are probably destined to be displaced by the processes starting from phlegms, especially as the rectification stills are readily convertible into plant suitable for dehydration by the azeotropic methods.

R. BRIGHTMAN.

General process of sulphonation. L. GAY, M. AUMÉRAS, and P. MION (Chim. et Ind., 1928, 19, 387—395).—Sulphonation of naphthalene with sulphuric acid of any concentration is readily effected by using an "auxiliary" liquid, sufficiently volatile, *e.g.*, carbon tetrachloride or ligroin, which is also of low miscibility with water, to eliminate the excess of water and prevent the concentration of the sulphuric acid falling below the minimal concentration (π) (cf. A., 1926, 605). Alternatively, a current of chemically inert gas may be used.

In the former case the liquid distilling from the sulphonation vessel is returned continuously after separation of the water eliminated. With equimolecular quantities of naphthalene and 94% sulphuric acid, a 91–94% yield of naphthalenemonosulphonic acids is obtained in 3–5 hrs. at 140–150°, using carbon tetrachloride as auxiliary liquid. About 6–7% of sulphones are formed and the reaction product contains no free sulphuric acid. Excess of naphthalene does not appreciably increase the formation of sulphones, the velocity of the reaction, $C_{10}H_7 \cdot SO_3H + C_{10}H_8 = (C_{10}H_7)_2SO_2 + H_2O$ being relatively small compared with that of sulphonation. Mercuric sulphate does not catalyse the formation of sulphone but catalyses the side-reaction between water and carbon tetrachloride, affording hydrogen chloride which is always formed to some extent, especially if the temperature exceeds 150°. Excess of sulphuric acid also promotes the formation of hydrochloric acid, but no sulphones are found in the reaction product, which consists of a mixture of mono- and di-sulphonic acids, together with some free sulphuric acid. Thus, with 2 mols. of sulphuric acid (94%) and 1 mol. of naphthalene, the reaction product after 3 hrs. (or 9½ hrs.) at 140–150°, contains 18.5% (9.3%) of free sulphuric acid, 39.5% (19.9%) of monosulphonic acids, and 33.5% (67.6%) of disulphonic acids. Similar results are obtained with ligroin or carbon dioxide as the auxiliary. In the latter case the elimination of water is practically complete in 12 hrs. Use of an inert gas in place of an auxiliary liquid offers advantages in the avoidance of side-reactions and in heat economy, even if the thermal efficiency of the apparatus is low. The proportions of isomeric naphthalene-mono- and di-sulphonic acids formed have not been determined, but experiments indicate that the isomerides are obtained at corresponding temperatures in approximately the same proportions as in ordinary sulphonation. Unsuccessful attempts have been made to adapt the methods of Guyot (B., 1919, 811 A) and of Ambler and Gibb (B., 1919, 405 A) to the sulphonation of naphthalene.

R. BRIGHTMAN.

Industrial preparation of perylene. A. CORBELLINI and G. AYMAR (Giorn. Chim. Ind. Appl., 1928, 10, 196–199).—Numerous experiments are described on the industrial preparation of perylene by the condensation of β -naphthol by known methods. The maximum yield of perylene, 19.3%, was given by the Hansgirk process (cf. B., 1921, 790), and the minimum, 13%, by the Marschalk process (cf. B., 1926, 869). No sensible improvement in the yield is obtained by the addition of sodium carbonate in the Pereira process. Contrary to the statement by Weitzenböck and Seer (A., 1913, i, 847), the condensation of β -dinaphthol to dihydroxyperylenes takes place in presence of aluminium chloride alone. The scanty yields obtained render it unlikely that the processes tested are capable of industrial application.

T. H. POPE.

"Contact substance" from petroleum distillates. VLASSENKO.—See II.

PATENTS.

Apparatus for the treatment of hydrocarbon compounds. E. R. HAMILTON (U.S.P. 1,671,423,

29.5.28. Appl., 17.11.24).—A closed container divided into concentric cells is used. The hydrocarbon enters the central compartment and flows alternately over and under transverse divisions in each cell in a more or less spiral course to the outermost cell, from which it is drawn off.

C. HOLLINS.

Manufacture of methyl alcohol. Soc. NAT. DE RECHERCHES POUR LE TRAITEMENT DES COMBUSTIBLES (F.P. 613,896, 31.7.25).—Carbon monoxide and hydrogen are passed at 160–180° and 150 atm. pressure over a catalyst consisting of precipitated copper hydroxide reduced in hydrogen at ordinary pressure; reduction begins at 140°, and the temperature must not exceed 450–500°.

C. HOLLINS.

Use of wood charcoal as catalyst in preparation of methyl alcohol from carbon monoxide and hydrogen. J. CAMPARDOU and J. VERGUES (F.P. 613,470, 3.3.25).—The gases are heated in a closed vessel at 300–400° in presence of wood charcoal.

C. HOLLINS.

Manufacture of methylene dichloride. I. G. FARBENIND. A.-G. (B.P. 283,119, 3.1.28. Ger., 3.1.27).—A mixture of methyl chloride (3 vols.) and chlorine (1 vol.) is passed at 120 litres per hr. through a tube (750–800 c.c.) at 360–380°. The product contains 92% of methylene dichloride, only 3% of chloroform, and traces of carbon tetrachloride, whereas methane yields greater proportions of the tri- and tetra-chloro-compounds. A catalyst may be used.

C. HOLLINS.

Manufacture of monocarboxylic acids. I. G. FARBENIND. A.-G. (B.P. 262,101 and Addn. B.P. 291,326, 22.11.26. Ger., [A] 27.11.25).—(A) Vapours of dicarboxylic acids with or without water vapour, or the corresponding anhydrides with water vapour, are passed over decarboxylating catalysts at 250–500°. Diluent moist gases may be added, and the gas mixture from the hydrogenation of unsaturated dicarboxylic anhydrides or from the oxidation of hydrocarbons etc. may be employed. Examples are: benzoic acid from phthalic anhydride; acrylic acid from maleic anhydride. (B) Oxides of zinc, cadmium, lead, bismuth, or mixtures containing one or more of these are selected as catalysts.

C. HOLLINS.

Manufacture of formic acid. A. SCHLOSS (G.P. 445,644, 18.9.24).—The kneading together of formate and acid and the distillation of the formic acid are combined in a single operation, under vacuum if desired, by use of a plant consisting of a two- (or more) chambered kneading machine with arms moving in opposite directions and sweeping the walls of the apparatus. The mass is thus evenly mixed and evenly heated, and the process occupies only one third of the usual time.

C. HOLLINS.

Purification of lactic acid. W. KLAPPROTH (B.P. 280,969, 22.11.27).—Crude 80% lactic acid (1000 pts.) is mixed with concentrated sulphuric acid (80 pts.) and anhydrous sodium sulphate (120 pts.) before extraction of the pure lactic acid content with ether.

C. HOLLINS.

Preparation of acetic anhydride. CONSORT. F. ELEKTROCHEM. IND., G.M.B.H., Assees. of R. MEINGAST and M. MUGDAN (G.P. 442,256, 25.6.25).—Acetic acid

vapour is heated to high temperatures, *e.g.*, 600°, in containers of graphite or calcined carbon preferably in presence of a catalyst (fused phosphates). Ordinary carbon has a destructive action on the acetic acid.

C. HOLLINS.

Manufacture of esters. I. G. FARBENIND. A.-G. (B.P. 265,233 and Addn. B.P. 292,059, 31.1.27. [A] Ger., 29.1.26).— β -Hydroxyethyl esters are obtained by heating ethylene oxide etc. with organic acids in presence of (A) an alkali salt of the acid or (B) a mineral acid, a mineral acid salt, or a small quantity of an alkali salt of another organic acid.

C. HOLLINS.

Manufacture of secondary amines. W. SCHILT (Swiss P. 117,162, 29.6.23).—Halogen acid salts of trialkylamines or arylalkylamines are heated with acylating agents until no more alkyl halide is split off, and the resulting acylated secondary amine is then hydrolysed. *E.g.*, dimethylaniline hydrochloride is heated, with gradual addition of acetic anhydride, to 170–180°. At about 155° methyl chloride begins to be evolved and acetic acid distils; after the reaction is complete, acetmethylanilide crystallises out on cooling, and may be recrystallised and hydrolysed. Hydrogen chloride, as gas or in acetic acid solution, may be added during the dealkylation.

C. HOLLINS.

Manufacture of $\gamma\delta\delta$ -tetramethylpentan- β -one [methyl $\beta\beta$ -dimethyl-*tert*-amyl ketone]. E. R. LOCQUIN (F.P. 592,216, 29.3.24).— $\beta\gamma\delta\delta$ -Tetramethyl- $\beta\gamma$ -glycol [$\beta\gamma$ -dihydroxy- $\beta\gamma\delta\delta$ -tetramethylpentane] is isomerised by concentrated sulphuric acid at 0° or by heating in alcohol with dilute 20% phosphoric, sulphuric, or oxalic acid under pressure at 150–160°, giving methyl $\beta\beta$ -dimethyl-*tert*-amyl ketone, $\text{CMe}_3\cdot\text{CMe}_2\cdot\text{COMe}$, m.p. 63–64°, b.p. 167–168° (*semi-carbazone*, m.p. 207–208°), a substitute for camphor as a plasticiser especially for cellulose acetate.

C. HOLLINS.

Manufacture of acyl peroxides. N. V. INTERNAT. OXYGENIUM MAATSCHAPPIJ "NOVADEL," Assees. of T. KROEBER (G.P. 441,808, 7.4.25).—The acyl peroxide (*e.g.*, benzoyl peroxide) is prepared in presence of finely-divided, preferably crystalline, substances, *e.g.*, quartz, silica gel, calcium hydroxide or carbonate, insoluble phosphates, oxides, carbonates, or silicates, or other microcrystalline compounds. Thus benzoyl chloride is added to a well-stirred mixture of hydrogen peroxide with 33% of sodium hydroxide and powdered quartz or with iron-free calcium hydroxide. The fine division of the product makes it very suitable for cleaning solids.

C. HOLLINS.

Separation of 2:4-dinitrophenol from admixture with picric acid. SOC. CHIM. DE LA GRANDE PAROISSE (F.P. 575,302, 7.1.24).—The mixed nitro-compounds are heated with water and iron turnings, whereby the picric acid dissolves as ferrous picrate and the dinitrophenol melts and may be removed as a cake after cooling. Picric acid is recovered by acidifying with sulphuric acid the ferrous picrate solution, and the dinitrophenol is purified by dissolving it in alkali hydroxide, filtering, and adding sulphuric acid.

A. R. POWELL.

Manufacture of naphthalene derivatives. O. Y. IMRAY. FROM I. G. FARBENIND. A.-G. (B.P. 291,965, 16.6.27).—Sulphonation of 6-hydroxy-2-naphthoic acid with concentrated sulphuric acid at 50–80° gives a separable mixture of 7-*sulpho*- and 4-*sulpho*-derivatives. With oleum at 25–50° the 4:7-*disulphonic acid* is obtained, which may be hydrolysed by heating with diluted sulphuric acid to give 6-hydroxy-7-*sulpho*-2-naphthoic acid.

C. HOLLINS.

Manufacture of derivatives of 2-[hydr]oxy-naphthalene-3-carboxylic acid amide [2:3-hydroxynaphthamide]. I. G. FARBENIND. A.-G. (B.P. 289,037, 4.1.28. Ger., 22.4.27).—2:3-Hydroxynaphthoic acid reacts with thiocarbimides to give *N*-substituted 2:3-hydroxynaphthamides (*cf.* Kay, A., 1894, i, 76); *e.g.*, phenylthiocarbimide gives the anilide in quantitative yield.

C. HOLLINS.

Production of wax-like polychloronaphthalenes. BRIT. DYESTUFFS CORP., LTD., J. B. PAYMAN, and W. GIBSON (B.P. 291,849, 292,056, and 292,058, 4.3.27).—(A) Chlorine is passed into molten naphthalene at 130–160° in presence of ferric chloride until 2 mols. have been absorbed, and the product is distilled at 230–260°/12–14 mm. The distillate is a hard, pale yellow, non-inflammable, insulating, wax-like substance, m.p. 131°, containing about 52.5% Cl, free from objectionable physical properties. (B) The naphthalene may be chlorinated in a solvent (carbon tetrachloride), and (C) the product may be mixed with α -chloronaphthalene in suitable proportions (4:1) to form a soft, crumbling solid which gives a good polish on wood, linoleum, etc.

C. HOLLINS.

Manufacture of wax-like mixtures of substances. BRIT. DYESTUFFS CORP., LTD., J. B. PAYMAN, and W. GIBSON (B.P. 292,057, 4.3.27).—The wax-like substances of B.P. 291,849 or 292,056 (preceding) are melted with sulphur to give a harder, whiter, and more porcelain-like mixture, m.p. about 111°, which adheres firmly to metallic and other surfaces.

C. HOLLINS.

Manufacture of side-chain-bearing polynuclear aromatic compounds or their sulphonic acids. I. G. FARBENIND. A.-G. (B.P. 267,132, 3.3.27. Ger., 3.3.26).—Polynuclear aromatic compounds (naphthalene) are heated at 60–95° with hydrogen sulphates of alkyl, aralkyl, or alicyclyl radicals, containing 3 or more carbon atoms, in presence or absence of condensing agents. Thus *n*-butyl alcohol is treated with chlorosulphonic acid to form *n*-butyl hydrogen sulphate, naphthalene is added, and the mixture is heated at 60–70° for condensation and finally at 95° for sulphonation.

C. HOLLINS.

Manufacture of α -naphthylamine-4:6:8-trisulphonic acid. E. HERTEL (Swiss P. 118,720, 12.12.25).—The reduction of 1-nitronaphthalene-4:6:8-trisulphonic acid with iron and acid gives improved yields in presence of copper, or salts of copper or nickel, or when a copper stirrer is used.

C. HOLLINS.

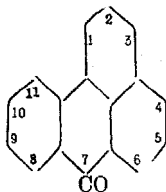
Manufacture of organic compounds containing a 1:3:5-triazine residue. GES. F. CHEM. IND. IN BASEL (Swiss P. 118,602–5, 7.10.24. Addns. to Swiss P. 103,430).—1:3:5-Triazine-2:4:6-tricarboxylic

C. HOLLINS.

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derivatives containing other replaceable groups may be used in place of the nitro-compounds. The following benzantrones are described (numbering as in annexed formula) :—3-methoxy-, m.p. 173°; 9-chloro-3-nitro-, m.p. 277°; 9-chloro-3-methoxy-, m.p. 262—263°; 3 : 9-dinitro-, m.p. 266°; 9-nitro-3-methoxy-, m.p. 318°;



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Acetone oils and wood spirit (F.P. 619,857).—See II.

PATENTS.

C. HOLLINS

Manufacture of vat dyes of the anthraquinone series. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 291,878, 10.3.27).—1-Amino-2-aldehydoanthraquinone is condensed with a halogenated anthraquinone in presence of sodium acetate and copper powder. The primary condensation products are converted into vat dyes (preferably after purification by precipitation from sulphuric acid) by boiling with dilute acid or better with alkali. From 1-amino-2-aldehydoanthraquinone and α -chloroanthraquinone an orange vat dye, identical with the acridone, $C_{14}H_6O_2 \cdot \begin{smallmatrix} NH \\ CO \end{smallmatrix} \cdot C_{14}H_6O_2$ of G.P. 268,219, is obtained: 1-chloro-5-benzamidoanthraquinone

(brownish-orange) and 1-chloro-4-benzamidoanthraquinone (dark blue-violet) give similar products.

C. HOLLINS.

Manufacture of new [thioindigoid] vat dyes containing sulphur. W. CARPMAEL. From I. G. FARBENIND A.-G. (B.P. 291,825, 3.2.27).—An isatin α -chloride or α -anil is condensed with a 4-alkyl-, aralkyl, or arylthiol derivative of α -naphthol to give vat dyes. Examples are: 5:7-dibromoisatin α -chloride with 4-hydroxy-1-naphthyl methyl or *p*-tolyl sulphide (greenish-blue).

C. HOLLINS.

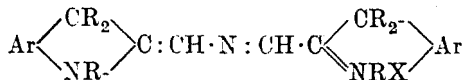
Preparation of soluble derivatives of indigoid vat dyes. I. G. FARBENIND. A.-G., Assees. of E. MÜNCH (G.P. 445,566, 14.3.25).—*N*-Acylated indigos etc. combine with sulphurous acid or sulphites to give water-soluble products which regenerate the starting materials by the action of cold ammonia, or are immediately hydrolysed to indigos by sodium hydroxide or hot ammonia. *E.g.*, *NN'*-diacetylindigo is heated for 30–60 min. under reflux with 40% sodium bisulphite solution and alcohol, more alcohol is added, and the yellow filtrate is evaporated in a vacuum to give a separable mixture of the white bisulphite compound of diacetylindigo and a deep yellow additive product. The *pyridine* salt of the bisulphite compound is formed when sulphur dioxide is passed into a suspension of *NN'*-diacetylindigo in aqueous pyridine; it may be converted into the sodium salt by action of sodium acetate and alcohol. Similar products are obtained from *NN'*-dibenzoylindigo, *NN'*-dicarbethoxyindigo, *N*-acetylindirubin, *N*-acetyl-3-indole-2'-thionaphthene-indigo (from *N*-acetylrisatin and thioindoxyl), 4:4'-dichloro-1:1'-diacetylindigo, *NN*-diacetylisoindigo (from isoindigo, acetic anhydride, and a little β -chloroethyl *p*-toluenesulphonate). The bisulphite compounds may be applied to wool from an acetic acid bath, and developed with ammonia.

C. HOLLINS.

Manufacture of new triphenylmethane dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 291,887, 11.3.27).—An *o*-hydroxy-acid (salicylic acid, hydroxytoluic acid) is condensed with 4-benzylaminobenzaldehyde-3-sulphonic acid in sulphuric acid at 30–35°, and the product oxidised in the usual manner to give a chrome-violet of good light-fastness.

C. HOLLINS.

Manufacture of new dyes from indoline bases. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 291,888, 11.3.27).—By the action of a nitrite or nitrous ester on 2-methylene-1:3:3-trialkylindoline or its salts (2-methyl-1:3:3-trialkylindoleninium salts) in presence of acetic anhydride, yellow to orange-red basic dyes, probably of the annexed formula, are obtained. Thus

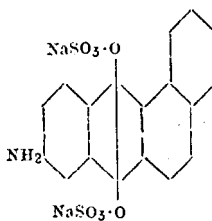


from 2-methylene-1:3:3-trimethylindoline, sodium nitrite, and acetic anhydride a light-fast, greenish-yellow dye for natural or artificial silk or tannin-mordanted cotton is prepared.

C. HOLLINS.

Manufacture of dyes of the anthracene series. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P.

291,851, 7.3.27).—Dyes of the indanthrone type are obtained by oxidation of a sulphuric ester of a leuco- β -amino- (or alkylamino-)anthraquinone having a free α -position *ortho* to the amino-group. The alkali salts of 2-amino-9:10-disulphatoanthracene, prepared by the action of chlorosulphonic acid and a tertiary base on leuco- β -acetamidoanthraquinone with subsequent hydrolysis, are oxidised with ferric chloride, or with ferric ammonium or sodium sulphates, to indanthrone. Substituted indanthrones are similarly obtained from 6(7)-chloro-, 6:7-dichloro-, and 6(7)-methyl derivatives of leuco- β -aminoanthraquinone, and from leuco- β -methylaminoanthraquinone. The leuco-ester salt of 9-amino- α -naphthanthraquinone (annexed formula) gives a green vat dye of similar type.



C. HOLLINS.

Manufacture of anthraquinone derivatives. BRIT. DYESTUFFS CORP., LTD., A. SHEPHERDSON, W. W. TATUM, and F. LODGE (B.P. 291,814, 13.12.26).—One or both alkyl groups are removed from 1:4-dialkylidiaminoanthraquinones by heating with 100% sulphuric acid at 180–220°, or with 100% sulphuric acid and boric acid at 130°. From 1:4-dimethyldiaminoanthraquinone a bluish-violet dye for acetate silk is obtained.

C. HOLLINS.

Manufacture of derivatives of *N*-dihydro-1:2 1':2'-anthraquinoneazine fast to chlorine. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 291,552, 18.3.27).—Indanthrone is chlorinated in 85–95% sulphuric acid containing a catalyst (nitrogen oxides) at about 50°, and the resultant azines are reduced to chlorinated indanthrones. The products may be fractionally precipitated by dilution. Dichloroindanthrone, prepared thus, is completely fast to chlorine.

C. HOLLINS.

Azo dyes. J. HALLER, Assr. to GRASELLI DYESTUFF CORP. (U.S.P. 1,671,422, 29.5.28. Appl. 23.6.27. Ger., 28.6.26).—Clear violet shades are obtained by coupling in substance or on the fibre a 2:3-hydroxynaphthoic arylamide (β -naphthylamide, 2:5-dimethoxyanilide) with a diazotised arylamine \rightarrow aminonaphthol methyl ether, *e.g.*, 1:7- or 1:8-.

C. HOLLINS.

Treatment of dyes. D. GARDNER (B.P. 291,827, 5.2.27).—Insoluble dyes, particularly vat dyes, and also soluble dyes such as purpurin, rhodamines, etc., are treated with 5 or 6 pts. of tetrachloride or tetrabromide of tin or titanium, with or without addition of acetic acid or other organic solvent soluble in water, at 15° or higher temperatures (60–75°), with the formation of water-soluble products which can be used for direct dyeing.

C. HOLLINS.

Manufacture of thiocarbazides and carbazides of the naphthalene series. G. M. DYSON, F. A. MASON, and A. RENSHAW, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,673,498–9, 12.6.28. Appl. 12.5.27. U.K., 25.5.26).—See B.P. 278,037; B., 1927, 902.

Production of benzanthrone derivatives. K. ZAHN and W. ECKERT, Assrs. to GRASELLI DYESTUFF

CORP. (U.S.P. 1,674,351, 19.6.28. Appl., 27.5.26. Ger., 27.6.25).—See B.P. 254,294; B., 1927, 837.

Anthraquinone derivatives (B.P. 272,469).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Properties of wool. L. MEUNIER and G. REY (J. Soc. Leather Trades' Chem., 1927, 11, 508—519).—When wool is subjected to ultra-violet light it loses its elasticity, fluorescence, and its property of double refraction. Its solubility in water and alkalis increases. Part of the sulphur in the wool molecule becomes more labile, and the sulphur liberated is converted into sulphurous acid. The curve for the swelling of wool shows minima at p_H 0.1 and 4.0—4.5 and a maximum at 1.5. Neither deaminated wool nor wool treated with quinone swells to the same degree as untreated wool. The second minimum is shifted towards p_H 5.0 by subjecting wool to ultra-violet light. Wool swells about equally in hydrogen sulphide and ammonia solutions, and to a lesser degree than in distilled water; the swelling is quite different from that produced by solutions of lime, baryta, or the strong alkalis, which seems to confirm Merrill's hypothesis (B., 1925, 18, 327). The primary amino-nitrogen content in wool is 0.57% as compared with 0.40% in silk fibroin, which shows less swelling at equal p_H values. D. WOODROFFE.

Hygroscopicity of cellulose esters. A. CAILLE (Chim. et Ind., 1928, 19, 402—406).—The relation between the hygroscopicity of nitrocellulose and its total sulphuric acid content is closely analogous to that previously observed between the sulphuric acid content and the absorption of basic dyes (B., 1926, 355). The hygroscopicity increases with the total sulphuric acid content, and samples containing the same amount of total sulphuric acid but different amounts of combined acid have practically the same hygroscopicity. Similar results were obtained with parchment paper which had been sulphurised by treatment with sulphuric acid, d 1.68, for 20 sec. (cf. B., 1927, 475). Examination of samples of cellulose acetate prepared as previously described (B., 1924, 937) shows that the fraction insoluble in acetone always contains less sulphuric acid than the original ester. Other things being equal, samples with the largest ratio of combined to total sulphuric acid are the most soluble in acetone and give the most viscous solutions. For a given sample of cellulose acetate, the viscosity of its solution in acetone decreases with the proportion of combined sulphuric acid in the sample. On fractional precipitation from acetone with aqueous acetone or water, cellulose acetate affords fractions differing widely in their sulphuric acid content, and experiments prove the existence of cellulose acetates soluble in 50% aqueous acetone. The sulphuric acid is therefore regarded as functioning otherwise than as a depolymerising agent, and in consequence of the dispersion in acetone the cellulose acetate is obtained in micelles of different sizes and varying sulphuric acid content. Bréguet's view that the largest micelles contain the highest proportion of combined sulphuric acid requires confirmation by ultrafiltration experiments. R. BRIGHTMAN.

Formation of poisonous gases in the manufacture of viscose artificial silk, and their removal. J. EGGERT (Chem.-Ztg., 1928, 52, 505—506).—Hydrogen sulphide is produced chiefly in the spinning process, during regeneration of the cellulose in the precipitating bath, and diffuses into the air. The production of only 1000 kg. of silk per day may cause the liberation of about 200 m.³ or 300 kg. in the air of the spinning room. Satisfactory designing of the spinning machines to prevent escape of the gas, admission of pure air from above, and removal of impure air from below the machines are recommended. Again, chlorine is liberated by acid-washing the yarn after bleaching it with hypochlorite liquors; this should be drawn off from the lowest part of the floor of the bleaching room and pure air admitted from above. B. P. RIDGE.

Saccharification of cellulose. LEONE and NOERA.—See XVII.

PATENTS.

Removal of fat from raw sheep's wool. I. G. FARBERIND. A.-G. (F.P. 623,103, 13.10.26).—The wool is treated with esters of carbonic acid either alone or admixed with other solvents for fats.

A. R. POWELL.

Manufacture of hollow viscose fibres. N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (G.P. 623,148, 14.10.26).—The viscose solution is spun directly into a bath containing 10% of ammonium sulphate and 12% of sodium carbonate, whereby part of the latter is absorbed by the fibres formed. The fibres then pass through a bath containing 15% of magnesium sulphate, 8% of sodium sulphate, 1% of zinc sulphate, and 9% of sulphuric acid. For a smooth fibre the material is sprayed with a 15% magnesium sulphate solution during its passage from the first to the second bath.

A. R. POWELL.

Colloid product [from viscose]. S. A. NEIDICH (U.S.P. 1,670,162, 15.5.28. Appl., 3.3.26).—An extruded colloid product comprising a solid core filament and an outer imperforate tubular sheath having a crinkled highly reflecting surface is produced by passing viscose filaments from spinnerets into a coagulating bath containing 16.5% of sulphuric acid and 0.8% of sodium phosphate. The bath effects almost complete removal of sulphur from the viscose and causes rapid coagulation of the outer skin and retarded coagulation of the interior.

A. R. POWELL.

Manufacture of a cellulose fibre product. I. F. LAUCKS, INC., ASSEES. of G. DAVIDSON, H. F. RIPPEY, C. N. CONE, I. F. LAUCKS, and H. P. BANKS (B.P. 270,335, 30.4.27. U.S., 3.5.26).—See U.S.P. 1,622,496; B., 1927, 438.

Funnels or thread guides for use in the manufacture of threads of artificial silk and the like. E. WAGNER (B.P. 292,216, 11.3.27).

Cleansing of funnels of cuprammonium artificial silk spinning machines. O. VON KOHORN [F. O. KOHORN & Co.], and A. PERL (B.P. 290,560, 28.1.28. Austr., 14.5.27).

Machine for beating, bleaching, refining, etc. of paper and like stock. U. KIRCHNER (B.P. 271,457, 12.5.27. Ger., 22.5.26).

Manufacture of figured paper, cardboard, etc. L. CLEMENS (B.P. 279,805, 27.9.27. Ger., 28.10.26).

[Conical-type] refining engines for use in pulp refining. W. G. FRASER (B.P. 292,228, 14.3.27).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Effect of oxycellulose on tests for mercerisation. E. RISTENPART (Textilber., 1928, 9, 577—579).—Two samples each of mercerised and non-mercerised cotton yarns containing relatively small and large amounts of oxycellulose were prepared by immersing suitable yarns for $\frac{1}{2}$ and 3 hrs. respectively in solutions of bleaching powder (d 1.007) at 50°, and their behaviour to several reagents commonly used in testing cellulosic materials were compared. Mercerised yarn suffers a greater loss of strength, has a greater solubility in caustic soda and a greater affinity for methylene-blue, and attains a purer white than unmercerised yarn when overbleached. Although the original mercerised and unmercerised cotton yarns had nearly the same copper value, the copper values of the resulting overbleached mercerised yarns were 50—60% higher than those of the overbleached unmercerised yarns. In application of the Lange test to the unmercerised (original, slightly bleached, and overbleached) and the corresponding mercerised yarns, the resulting blue (iodine) colorations of the yarns resisted discharge by washing for 3, 2.3, 2, 7.5, 6.5, and 3.5 min. respectively. Mercerisation of overbleached cotton can be detected by microscopical examination of the fibres, but if oxycellulose is present the Lange test cannot be used for determining the degree of mercerisation.

A. J. HALL.

Organic peroxides as bleaching agents. HOOFT.—See XII.

PATENTS.

Dyeing with vat dyes. D. GARDNER (B.P. 291,828, 5.2.27).—Textile fibres, particularly wool and silk, are dyed in neutral or slightly acid solutions prepared by dissolving vat dyes in water-soluble organic solvents (e.g., acetic acid, alcohols, ketones, esters, aldehydes, etc.), reducing the dyes by the addition of titanous chloride, and then suitably diluting with water. Alternatively, the dye liquor is prepared by the addition of titanous chloride to the product obtained by treating the vat dyes with anhydrous tetrachlorides or tetrabromides of metals of the fourth group in the periodic system, e.g., titanium tetrachloride. It is preferable to dye artificial silk and vegetable fibres from a neutral bath, the organic solvent being replaced by an aqueous solution of a carbohydrate such as dextrose.

A. J. HALL.

Development of vat dyes [indigosols] on the fibre. I. G. FARBENIND. A.-G., Assees. of K. JELLINEK (G.P. 445,251, 23.1.26. Addn. to G.P. 441,984).—Oxalic acid is used, in place of the formic acid of the prior patent, to liberate nitrous acid for development of indigosol prints.

C. HOLLINS.

Dyeing of acetate silk. I. G. FARBENIND. A.-G. (B.P. 274,823, 24.5.27. Ger., 24.7.26).—Acetate silk is dyed with unsulphonated azo dyes containing a di-(hydroxyethyl)amino-group. Thus di-(β -hydroxyethyl)-

aniline is coupled with diazotised *p*-nitroaniline (yellow) or 3 : 4 : 5-trichloroaniline (orange); di-(β -hydroxyethyl)-*m*-toluidine, m.p. 71—72°, with diazotised *p*-nitroaniline (dark yellowish-red) or 2 : 4-dinitroaniline (bluish-bordeaux).

C. HOLLINS.

Dyeing, printing, or stencilling of materials made of or containing cellulose derivatives. H. DREYFUS (B.P. 291,118, 23.11.26).—One or more thiocyanato-, cyano-, thiocarbimido-, carbimido-, ureido-, or thioureido-groups are introduced into dyes or dye components by known methods, and the products are used for dyeing acetate silk etc. Examples are: 1-acetamido-4-thiocyananthraquinone (yellow), 1-anthraquinonylthiocarbamide (orange), *N*-ethyl- or *N*-allyl-*N*'-1-anthraquinonylthiocarbamide (orange); 2 : 4-dinitroaniline \rightarrow 5-thiocyanosalicylic acid (brownish-yellow); aniline \rightarrow α -naphthylamine \rightarrow *o*-hydroxyphenylcarbamide (golden-yellow); *m*-aminophenylcarbamide \rightarrow aniline, coupled on the fibre with *m*-hydroxyphenylcarbamide (reddish-yellow); 2 : 2'-dinitro-4 : 4'-dithiocyanodiphenylamine (yellow); 3 : 3'-dinitro-4 : 4'-diureidodiphenyl (lemon-yellow), and 3 : 3'-dinitro-4 : 4'-diureidodiphenylmethane (greenish-yellow). The usual methods of dyeing are employed.

C. HOLLINS.

Manufacture of colour-printed fabrics. J. MORTON (B.P. 292,415, 22.11.27).—Colour-printed patterned fabrics are prepared by applying colours by means of plain (not engraved) printing rollers or blocks to fabric having figures or designs produced therein by jacquard mechanism, such figures containing dyed yarns or yarns pretreated so that they are capable of resisting or modifying the colour printed upon them.

A. J. HALL.

Printing pastes for textile materials. R. GAUNT, C. L. WALL, and BLEACHERS' ASSOC., LTD. (B.P. 292,441, 27.2.28).—Glycols having not less than three and not more than five carbon atoms are particularly suitable for addition to printing pastes containing basic dyes, since they are excellent solvents for the latter, e.g., Acridine Orange DH and New Blue B.

A. J. HALL.

Weighting of silk. E. ELÖD, Assr. to R. KOEPP & Co. (U.S.P. 1,674,356, 19.6.28. Appl., 2.11.22. Ger., 14.11.21).—See G.P. 389,813; B., 1924, 708.

Preparation of [immunised] cotton fibres. P. KARRER, Assr. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,673,627, 12.6.28. Appl., 14.7.27. Switz., 11.3.25).—See B.P. 249,842; B., 1927, 71.

[Machine for elastic] mercerisation of fabrics. F. THIES (B.P. 291,130, 18.2.27).

Wet treatment of hanks of textile material. O. VON KOHORN and A. PERL (B.P. 274,885, 20.7.27. Ger., 20.7.26).

[Continuous] washing of silk piece goods. Soc. CLERTIN (B.P. 284,674, 3.2.28. Fr., 3.2.27).

Coloured nitrocellulose (B.P. 291,539).—See XIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Production of concentrated nitric acid by oxidation of ammonia under pressure. G. FAUSER (Giorn. Chim. Ind. Appl., 1928, 10, 183—195).

results of experiments on the oxidation of ammonia in presence of platinum as catalyst, under various pressures and with different velocities of, and different proportions of air or oxygen in, the mixed gases, show that the use of increased pressure is attended by considerable advantages. The velocity of oxidation of the nitrous vapours increases rapidly as the pressure is raised, and the absorption occurs far more readily, so that much smaller towers may be used. The practical difficulties prove by no means insurmountable. Joints present little trouble, since at 5 atm. pressure the cross-section of the piping is reduced to one sixth, and chromium-steels appear to be suitable construction materials. The small amount of extra energy required when pressure is used is largely compensated for by the diminished cost of the plant and the greater concentration of the resulting acid. A saving may be effected of 1 ton of steam, required for pre-concentration, per ton of acid produced, and the final concentration is simplified. If the production of nitrogen is not desired, and if a sufficient amount of by-product oxygen is available, the residual gases may be sent directly, without re-compression, to the gas producers. In this way the alkaline absorption is avoided and it is necessary only to compress the ammonia-oxygen mixture in the closed circuit; the cost of compression is about one third of that involved in compressing ammonia-air mixtures. In an industrial plant with four converters, each having a capacity of 2500 kg. of 100% nitric acid per day, acid of d 1.436 has been obtained. This acid could be concentrated by distillation through a rectifying column, or, if dehydration is effected by distillation with sulphuric acid, only one fourth as much sulphuric is required as is necessary when acid of d 1.33 is treated.

T. H. POPE.

Activated silica gel (sulphosil). B. TYTSCHININ and W. TOKMANOV (Neftjanoe Chozjajstvo, 1927, 12, 414—415; Chem. Zentr., 1927, II, 1524).—Instead of treating petroleum products etc. with concentrated or fuming sulphuric acid, and then with an adsorbent, the oil is treated with silica gel moistened with sulphuric acid (sulphosil). Silica gel is dried at 300° and, while still hot, mixed with concentrated sulphuric acid at 200—250° or with fuming acid at about 50°. The gel can take up 2—4 pts. of the concentrated acid and 0.6 pt. of sulphur trioxide; usually, gel containing only 0.2 pt. of oleum is used. Sulphosil is hygroscopic and loses its activity in contact with moist air; it also reacts with alcohol, benzene, or chloroform, but not with benzine.

W. S. NORRIS.

Ammonia-soda process: physicochemical action of the carbon dioxide tower. E. I. ORLOV (Ukraine Chem. J., 1928, 3 [Tech.], 1—29).—In the upper part of the carbon dioxide tower used in the Solvay process, conditions are such that sodium bicarbonate and carbonate may crystallise out. This is shown experimentally by saturating an appropriate sodium chloride-ammonia solution with carbon dioxide and analysing the solution and salts crystallising out. Means of avoiding this side reaction are suggested, and, on the basis of a study of phase conditions, certain faults present in the Donetsk and Berezniki factories are pointed out and the necessary alterations suggested.

R. TRUSZKOWSKI.

Determination of barium hydroxide. E. VON DRATHEN (Chem.-Ztg., 1928, 52, 518).—Titration of barium hydroxide with hydrochloric acid gives results in close agreement with those obtained gravimetrically. Lower values, which may show a deficiency in BaO of as much as 1%, are obtained when using sulphuric or oxalic acid, owing to the absorption of baryta solution by the precipitated sulphate or oxalate. F. R. ENNOS.

Titration of bleaching powder with nitrite solution. Z. KERTÉSZ (Z. anal. Chem., 1928, 74, 105—108).—In the titration of bleaching powder, or of the bleach liquors obtained from it, by the nitrite method, an excess of sodium hydrogen carbonate should be added if much free lime is present. In the titration of bleach liquors containing free sodium or potassium hydroxide, boric acid must be added in excess of that required to saturate the alkali.

A. R. POWELL.

Toxicities of arsenicals and fluorine compounds. MARCOVITCH.—See XXIII.

PATENTS.

Purification of silica gel. SILESIA VER. CHEM. FABR., and P. SCHLÖSSER (G.P. 447,139, 16.4.26).—The silica gel obtained by passing into water silicon fluoride obtained in the manufacture of superphosphates is purified by heating it in a current of steam to expel organic vapours, boiling an aqueous suspension of the product with potassium permanganate until the solution remains pink, and, finally, digesting with sulphur dioxide.

A. R. POWELL.

Purification of sodium sulphate. M. A. PURDY, Assr. to PACIFIC DISTRIBUTING CORP. (U.S.P. 1,673,471, 12.6.28. Appl., 7.2.23).—Anhydrous sodium sulphate is crushed wet in a saturated solution of sodium sulphate, and the crushed salt is passed backwards through a continuous flow of sodium sulphate solution of gradually decreasing strength.

A. R. POWELL.

Manufacture of globular sodium hydrogen sulphate. C. P. LINVILLE and C. E. MENSING, Assrs. to CALCO CHEM. CO. (U.S.P. 1,671,866, 29.5.28. Appl., 18.12.24. Renewed 17.9.27).—Molten sodium hydrogen sulphate is fed into the centre of a flat pan with outwardly sloping sides while the pan is rotated at a high speed within a deep chamber, so that the droplets of liquid which are thrown out by centrifugal force solidify in globules before they reach the floor of the chamber.

A. R. POWELL.

Apparatus for recovering salts from hot solutions, e.g., potassium chloride liquors. F. KRUPP GRUSONWERK A.-G. (G.P. 447,059, 12.10.22).—The hot liquor is projected through a centrally disposed horizontal spray into a flat circular vessel provided with a series of revolving rakes which continuously move the deposited salt towards a central discharge opening in the vessel.

A. R. POWELL.

Manufacture of solid tripotassium phosphate. I. G. FARBENIND. A.-G., Assees. of E. JÄNECKE (G.P. 446,110, 21.5.26).—A concentrated solution containing potassium and phosphate ions in the correct proportions is saturated with ammonia; the tribasic potassium salt is precipitated in the form of a crystalline meal.

A. R. POWELL.

Manufacture of potassium sulphate and ammonium chloride. N. SEVRIN (F.P. 622,684, 8.2.26).—A mixture of potassium chloride and ammonium sulphate in stoichiometrical proportions is added to such a quantity of water below 35° that the potassium sulphate formed by double decomposition is almost completely precipitated while the ammonium chloride is retained in solution. A. R. POWELL.

Manufacture of ammonium nitrate and blanc fixe. WOLFF & Co., and F. FROWEIN (G.P. 445,668, 26.5.25).—A mixture of ammonium sulphate and barium carbonate is stirred into a solution containing more than 15% of nitric acid, the resulting barium sulphate is separated, and the solution evaporated to obtain ammonium nitrate. A. R. POWELL.

Preparation of antimony [penta]sulphide. H. NUHN and L. H. BLOOD, Assrs. to ANTIMONY PRODUCTS CORP. (U.S.P. 1,671,203, 29.5.28. Appl., 27.7.22).—Finely-ground stibnite is digested with the requisite quantities of sodium sulphide and sulphur in hot water to obtain a solution of sodium thioantimoniate. This solution is diluted to d 1.13 and sprayed into a chamber through which sulphur dioxide is passed. The resulting mixture of antimony pentasulphide and sulphur is separated from the sodium thiosulphate solution formed at the same time, and, after drying, may be used directly for vulcanising rubber. By treating the thiosulphate solution with antimony trichloride "antimony crimson" is obtained, with the regeneration of sulphur dioxide for the first operation. A. R. POWELL.

Manufacture of calcium cyanamide. G. E. COX, Assr. to AMER. CYANAMID CO. (U.S.P. 1,674,466, 19.6.28. Appl., 17.6.25).—Ground calcium carbide is covered with a layer of comminuted kieselguhr and caused to react with nitrogen. H. ROYAL-DAWSON.

Preparation of alumina [from bauxite, clays, etc.]. J. C. SEAILLES (B.P. 283,509, 11.1.28. Fr., 11.1.27. Addn. to B.P. 277,697; B., 1928, 230).—Calcium aluminate is made by adding at least 4 mols. of lime for each mol. of alumina in the ore, and, optionally, 2–3 mols. of lime for each mol. of silica. The mixture is then ground with water in a colloid or other mill, and subjected to prolonged treatment in an autoclave to fix the silica. The calcium aluminate is washed to remove lime, treated with sodium carbonate solution or with caustic soda solution in presence of carbon dioxide, and the aluminium hydroxide is recovered from the sodium aluminate formed. W. G. CAREY.

Manufacture of anhydrous aluminium chloride. N. C. CHRISTENSEN (U.S.P. 1,673,495, 12.6.28. Appl., 7.6.23).—Aluminium is heated, in the presence of the chloride of a metal having less affinity for chlorine than aluminium at high temperatures, to a reacting temperature, the aluminium chloride being then separated from the residue. H. ROYAL-DAWSON.

Crystallisation of chrome alum. KÖNIGSBERGER ZELLSTOFF-FABR. U. CHEM. WERKE KOHLYT A.-G., and E. SCHLUMBERGER (G.P. 447,070, 21.4.26).—A solution of chromic and potassium sulphates in the correct proportions is acidified with nitric acid and treated with formic acid or a formate to promote more rapid crystallisation of the alum. A. R. POWELL.

[Separation of] asbestos [from rock]. H. SIMON, LTD., and F. R. JOLLEY (B.P. 291,864, 8.3.27).—The crushed and screened material is fed into a washing trough containing a worm-type conveyor and having a slot through which heavy material falls, a further separation being effected through a grid or screen in the channel along which the rock travels.

W. G. CAREY.

Manufacture of magnesium phosphate. VER. F. CHEM. U. MET. PROD. (G.P. 447,393, 13.3.25).—Tricalcium phosphate is boiled with magnesium chloride and hydrochloric acid until complete conversion into magnesium phosphate takes place. A. R. POWELL.

Manufacture of phosphotungstomolybdenum compounds and of lakes therefrom. W. CARPMAEL. From I. G. FARBEIND. A.-G. (B.P. 292,253, 17.3.27).—Compounds obtained by the action of reducing agents, e.g., sulphur dioxide, sodium bisulphite or hyposulphite, or glucose, on complex acids having the formula $(a\text{WO}_3 + b\text{MoO}_3) \cdot \text{P}_2\text{O}_5 \cdot 3\text{H}_2\text{O}$, where $a + b = 24$ or 18 (cf. G.P. 445,151; B., 1928, 123), are used in the production of lakes from basic dyes or acid dyes containing free or substituted amino-groups in addition to sulpho-groups; e.g., Rhodamine B yields a red lake fast to light. L. A. COLES.

Base-exchange bodies. SELDEN CO., Asses. of A. O. JAEGER (B.P. 279,466, 19.10.27. U.S., 19.10.26).—Base-exchange materials for use as catalysts are prepared by treating a solution of an alkali silicate with the salt of a metal capable of becoming part of the non-exchangeable nucleus, then with a salt of a metal acid, e.g., vanadic, molybdic, tungstic acid, etc. The gelatinous mass is stirred, filtered, and dried; it may then be subjected to a base-exchange process or heated in a suitable oxidising or reducing atmosphere to induce the desired catalytic properties. If desired, the zeolite may be incorporated with inert porous material either during or after formation. A. R. POWELL.

Catalytic process for the manufacture of hydrogen. R. J. A. GRENIER (B.P. 271,523, 23.5.27. Belg., 22.5.26).—Carbon monoxide and steam or water-gas and steam are passed over wood charcoal at 375°, yielding a mixture of hydrogen and carbon dioxide with a trace only of carbon monoxide.

W. G. CAREY.

Production of hydrogen-nitrogen mixtures. M. CASALE-SACCHI (B.P. 292,342, 7.6.27).—Liquid air is distilled to give pure nitrogen, and the oxygen (containing 2–3% N) is used for the incomplete combustion of hydrocarbons in the presence of steam. The carbon monoxide formed is catalytically converted into carbon dioxide and is removed, and the water is separated from the hydrogen by cooling; the pure nitrogen is then added to the hydrogen in suitable amounts, having regard to the nitrogen content of the oxygen used, for the synthesis of ammonia. W. G. CAREY.

Liquefying and separating the constituents of gaseous mixtures at low temperatures. L'AIR LIQUIDE SOC. ANON. POUR L'ÉTUDE ET L'EXPLOIT. DES PROC. G. CLAUDE (B.P. 281,675, 30.11.27. Fr., 4.12.26).—Oxides of nitrogen are removed from gas mixtures to be used in the manufacture of synthetic ammonia by

passing them prior to compression through a red-hot tube so as to decompose the nitrogen oxides into the constituent gases. Sulphuric acid used in the purification of gas mixtures is freed from oxides of nitrogen by treating it with a current of crude coal gas or with gases containing hydrogen sulphide. The presence of nitrogen oxides in gases to be liquefied may cause explosions if acetylene or other hydrocarbons are also present. A. R. POWELL.

Isolation of hydrogen cyanide from gaseous mixtures. I. G. FARBENIND. A.-G., Assocs. of A. MITTASCH and W. MICHAEL (G.P. 444,502, 2.11.22).—Carbon dioxide is removed as ammonium carbamate etc. by addition of ammonia, or sufficient ammonia is added to remove hydrogen cyanide also and the mixture is fractionated. In the synthesis from carbon monoxide and ammonia, excess of the latter sufficient to combine with the carbon dioxide may be used. C. HOLLINS.

Stabilisation of liquid hydrogen cyanide. DEUTS. GOLD- U. SILBER-SCHNEIDENSTALT, VORM. ROESSLER (G.P. 443,741, 3.7.25).—Those metals (or their alloys or compounds) which form complex compounds with ammonia or amines stabilise liquid hydrogen cyanide. Preferably they are used in thin plates, gauze, or powder (e.g., nickelled or coppered sheets etc.). Traces of organic or inorganic acids may be added. C. HOLLINS.

Manufacture of hydrogen sulphide or sulphides from sulphur. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 292,186, 10.1.27).—Sulphur or inorganic compounds yielding it, e.g., polysulphides, are treated under pressure above 100° but below dull redness with hydrogen, carbon monoxide, formic acid, or formates, preferably in the presence of catalysts, e.g., silica gel, alumina, active charcoal, or metals of the eighth group of the periodic system or their oxides, hydroxides, sulphides, or carbonates. L. A. COLES.

Recovery of sulphur from iron sulphides. R. F. BACON (U.S.P. 1,672,924, 12.6.28. Appl., 13.4.27).—Iron sulphides consisting mainly of the monosulphide are treated with sulphur dioxide so as to liberate sulphur and produce iron oxide. F. G. CLARKE.

Removal of free chlorine and bromine from fluid mixtures. BRIT. DYESTUFFS CORP., LTD., J. B. PAYMAN, and H. A. PIGGOTT (B.P. 292,307, 27.4. and 4.5.27).—The free halogen is removed from hydrogen chloride or bromide, either gaseous or in solution, by treatment with naphthalene, anthracene, solid olefines, or other solid hydrocarbon capable of forming additive compounds with the halogen. C. HOLLINS.

Extraction of the constituents of marine algæ. G. J. B. CHAMAGNE (B.P. 275,999, 11.8.27. Fr., 11.8.26).—The algæ are carbonised, finely ground, and burned (with the addition of ordinary powdered fuel, if necessary) in suitable plant provided with heat regenerators. The volatilised salts are precipitated electrically, dissolved in water, and the chlorides crystallised out from the cooled solution. The iodides and bromides are recovered from the mother-liquor by known methods. W. G. CAREY.

Concentration and distillation of solutions of hydrogen peroxide. I. G. FARBENIND. A.-G. (B.P.

264,535, 17.1.27. Ger., 18.1.26).—A dilute solution of hydrogen peroxide may be concentrated, or a concentrated solution purified, by passage in a finely-divided state or in thin layers through tubes or down a tower filled with non-catalytic fillers, e.g., glass or porcelain balls, while a strong current of gases travels in counter-current to the liquid. External heating may be applied or dried and preheated gases may be employed, using them again after drying for further treatment of the peroxide. W. G. CAREY.

Apparatus for making leaden powder [litharge]. C. A. HALL (U.S.P. 1,675,345, 3.7.28. Appl., 29.3.24. Renewed 2.12.27).—See B.P. 251,449; B., 1926, 584.

Separation of ingredients from an alkaline mixture containing oxysalt of arsenic and/or oxysalt of tin. H. HARRIS (U.S.P. 1,674,642, 26.6.28. Appl., 22.11.23. U.K., 4.12.22).—See B.P. 213,626; B., 1924, 522.

Purification of barium carbonate. J. E. MARWEDEL and J. LOOSER, Assrs. to RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (U.S.P. 1,673,985, 19.6.28. Appl., 2.12.26. Ger., 4.1.22).—See G.P. 427,223; B., 1926, 630.

Production of [soluble] carbonates. M. BUCHNER, Assr. to A. F. MEYERHOFFER (U.S.P. 1,675,786, 3.7.28. Appl., 18.6.25. Ger., 14.6.24).—See B.P. 235,588; B., 1925, 714.

Production of hydrogen. F. G. LILJENROTH and M. LARSSON, Assrs. to PHOSPHORUS-HYDROGEN CO. (U.S.P. 1,673,691, 12.6.28. Appl., 26.5.25. Swed., 29.5.23).—See G.P. 409,344; B., 1925, 448.

Acid-proof structures (B.P. 292,334).—See IX. **Complex mercury compounds** (B.P. 292,245).—See XXIII.

VIII.—GLASS; CERAMICS.

Silica bricks made without added bond. W. HUGILL and W. J. REES (Trans. Ceram. Soc., 1928, 27, 97—103).—Bricks were made from ganister and quartzite without the addition of a bond, and also, for purposes of comparison, with 2% of lime as bond. They were fired in an industrial down-draught kiln to cone 16—17. The percentage linear expansion in the kiln, compressive strength, powder and bulk density, and volume porosity were determined on the fired bricks. In the bricks without bond the degree of quartz conversion was greater than in the lime-bonded bricks, but the inversion was almost entirely to cristobalite. The addition of the bond facilitated tridymite formation, but did not accelerate the rate of quartz inversion. F. SALT.

Use of manganese in the manufacture of face bricks. G. E. SEIL and H. A. HEILIGMAN (J. Amer. Ceram. Soc., 1928, 11, 241—248).—Manganese dioxide is used either to change the background colour of a clay brick or to produce a speckled material. A grey colour can be produced without a colorant by soaking in a reducing atmosphere. 0.5% of air-floated manganese ore will deepen the colour under the same firing conditions. In order to obtain the greatest effect of the manganese ore in the manufacture of speckled bricks

it is essential to have a reducing atmosphere during the later stages of firing. Overfiring causes the manganese spots to pit the brick. In the case of clays maturing below 1000° the addition of a colorant has no effect, since the temperature never reaches the point at which the colorant fuses with the clay. The tinctorial effect of any addition is proportional to its exposed surface. It is essential to control the particle size of the colorant. A. T. GREEN.

Influence of different felspars on the "freezing" behaviour of cones. E. ORTON, JUN., and J. F. KREHBIEL (J. Amer. Ceram. Soc., 1928, 11, 215—223).—The "freezing" of a ceramic body is defined as its increased resistance to fusion consequent on an interruption of its heat-treatment in the vitrification zone, usually by chilling. Divergency in the behaviour of German and certain American pyrometric cones, which give identical values under normal firing conditions, is exhibited when they are subjected to a freezing treatment. Thus, a "frozen" American cone gave a value of 1295°, a raw cone from the same batch 1251°, whereas two comparable German cones each gave 1260°. It is shown that this effect occurs more seriously with the use of Hybla felspar instead of Swedish spar in the cone mixture. The kaolin used also has some influence. No evidence of crystallisation in the "frozen" cones could be found. The observed differences, in the main, related to the degree of completeness with which the felspar particles had dissolved in the glass. No explanation of the effect is put forward. A. T. GREEN.

Effect of heat on the crystalline break-up of kaolin. J. F. HYSLOP and H. P. ROOKSBY (Trans. Ceram. Soc., 1928, 27, 93—96).—Samples of kaolin were heated in an expansion furnace at the rate of 12° per min. to various temperatures between 600° and 1300°. The fired specimens were powdered and their X-ray patterns examined. The results indicated that kaolin breaks down at 550° to a crystalline phase (α), which is stable to 870°. At 870° the α -phase breaks down and two phases, β and either sillimanite or mullite, appear. The β -phase disappears at 1060°, mullite and silica remaining. Further heating eliminates the β -phase and mullite becomes well defined. The α - and β -patterns have not yet been identified. F. SALT.

Terra-cotta slips. E. C. HILL (J. Amer. Ceram. Soc., 1928, 11, 260—263).—The original body used contained 60% of clay and 40% of grog through 16-mesh. 70% of the clay content was made up of New Jersey tight-firing clay. No satisfactory slips for this body were obtained. The felspar-clay slips had a tendency to crack; slips with 50% of clay or less crazed, whilst others gave an unsatisfactory colour. Crazing decreases with increase in the content of clay or of Cornish stone. A similar body containing 60% of the New Jersey tight-firing clay was made up. A number of slips satisfactory for this body were obtained. The slip containing 43.7% of clay, with equal amounts of felspar and Cornish stone, is probably the safest slip developed, since it is equally removed from crazing and cracking on the one hand and from immaturity on the other. A. T. GREEN.

Gas-producer operation. WINDETT.—See II.

PATENTS.

Manufacture of glass opaque to X-rays. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST.-GOBAIN, CHAUNY, ET CIREY (B.P. 284,648, 26.1.28. Fr., 2.2.27).—A glass effective for use as a protective screen from X-rays contains the oxides of lead and barium in the proportion given by the formula $B \times 1.005(P + 350) = 110$, B being the percentage of barium oxide and P that of lead oxide in the glass. A. COUSEN.

Manufacture of a readily-fusible glaze or enamel. PATENT TREUHAND GES. F. ELEKTR. GLÜHLAMPEN, ASSEES. OF H. NACHOD (G.P. 445,736, 5.12.24).—To the usual enamel mixture of boric acid, silica, zinc oxide, and 40—60% of lead oxide is added 8—16% of an alkali silicofluoride consisting wholly or partly of the lithium salt. The coefficient of thermal expansion of the product is lower than that of the lithium-free glazes. A. R. POWELL.

Tunnel kilns. L. MELLERSH-JACKSON. FROM AMER. ENCAUSTIC TILING CO., LTD. (B.P. 291,504, 28.2.27).—The firing chamber of a tunnel kiln is provided with a stationary floor, the under side of which is exposed directly to the hot gases. This floor forms part of a muffle, which extends the full length of the tunnel and is heated at the sides, top, and bottom. The ware to be fired is placed on slabs of refractory material, e.g., carborundum; each loaded slab is placed on an exterior extension of the muffle floor, and is pushed into the kiln by means of a plunger. All the slabs are in contact, edge to edge, and also in direct contact with the floor of the muffle. Alternatively, a thin layer of sand may be placed between the stationary floor and the slabs. F. SALT.

Kilns for burning clay products, glassware, and pottery. H. WEBSTER, SEN. (B.P. 291,482, 27.1.27).—A kiln is provided with two series of fire grates, one of which is arranged to fire with an up-draught, and the other with a down-draught. The whole of the contents of the kiln are fired with the draught first in one direction and then in the other, only one series of grates being in operation at a time. In a series of such kilns waste heat from either series of grates in any one kiln can be passed by a system of flues to any other kiln of the series with an up-draught or down-draught, or to dryers. F. SALT.

Drying kilns for ceramic ware. BOMKESSEL & CO. M.B.II. (G.P. 445,817, 13.6.23).—The units of a series of drying kilns are coupled to one another by a series of flues closed by valves which permit of the hot air and flue gases being led as required from one chamber to another or to the stack. The air used for drying is preheated. A. R. POWELL.

Process of forming ceramic articles and the like. Manufacture of ceramic products. Apparatus for making ceramic articles. T. S. CURTIS (B.P. 291,523—5, 3.3.27).—(A) A ceramic body is prepared in a "soft mud" consistency, a small percentage of an electrolyte (sodium silicate) is added, and the mixture thoroughly mixed in a blunger until the body has reached the consistency of a casting slip, such consistency being maintained as long as the mixture is agitated. When

agitation ceases, the slip consistency disappears. The body is cast while in the slip condition, and means are provided for maintaining the agitation while the slip is in the mould or being conveyed thereto. When agitation is discontinued, the body hardens quickly and can be removed promptly from the mould. Non-plastic, refractory bodies can be cast by this method. Air may be removed from the body mixture by treatment under a vacuum, and the casting operation may be carried out under air pressure. (B) A ceramic body formed of fibrous mullite crystals and a binder is obtained as the product of firing purified natural cyanite with additional alumina in splintery form. The latter substance is in the form of artificial corundum, obtained from alunite, and the binder may be a siliceous material, or magnesia and lime. The body mixture is practically free from clay, and articles are formed therefrom by the process outlined above. Maximum strength etc. is obtained by firing the ware to Orton cone 32. (C) Suitable apparatus is described. F. SALT.

Production of shaped metal-containing ceramic and like articles. M. HAUSER (B.P. 270,300, 26.4.27. Switz., 28.4.26).—Mixtures of ceramic materials with one or more metals or alloys, *e.g.*, iron, copper, nickel, ferromanganese, ferrosilicon, etc., are shaped and fired under such conditions that the free metal is present in the finished article. Oxidation of the metal or alloy is prevented by the use of a composition which sinters at a relatively low temperature, by covering the article with a readily fusible glaze, or by firing in a non-oxidising atmosphere. Metals of high m.p., *e.g.*, tungsten and tantalum, are used in the production of refractory products containing magnesia and/or zirconia.

L. A. COLES.

Refractory material. J. G. DONALDSON and H. L. COLES, ASSRS. to GUARDIAN METALS CO. (U.S.P. 1,673,106—7, 12.6.28. Appl., [A] 16.1.25, [B] 3.7.26).—(A) The material consists of at least 70% of zirconia, less than 30% of zirconium silicate, and carbon, the last-named being dissolved in the silicate. (B) The refractory contains more than 85% of zirconia, less than 15% of silica, and less than 10% of carbon, the carbon being dissolved in the silicate formed during fusion of the oxides.

H. ROYAL-DAWSON.

Manufacture of refractory articles. G. S. DIAMOND, ASSR. to ELECTRIC REFRACTORIES CORP. (U.S.P. 1,674,961, 26.6.28. Appl., 23.9.26).—A mixture of magnesium oxide, crystalline graphite, a carbon binder, and a glaze is formed into the desired shape and baked; the product is again coated with glaze and rebaked.

H. ROYAL-DAWSON.

Manufacture of abrasives. H. BERCKEMEYER (G.P. 447,051, 18.2.26).—Loam is mixed with a small quantity of a flux, *e.g.*, sodium chloride, and the mixture is heated in a gas-fired furnace with a regulated air supply until it begins to melt. The product is cooled and pulverised.

A. R. POWELL.

Manufacture of abrasives. W. MARTINOV and H. BERCKEMEYER (G.P. 447,391, 28.2.25).—Loam which has been clinkered at 1500° is ground with sand and a flux, *e.g.*, sodium chloride, and the mixture is fused, cooled, and pulverised.

A. R. POWELL.

Oven for burning refractories etc. H. KOPPERS, ASSR. to KOPPERS DEVELOPMENT CORP. (U.S.P. 1,674,985, 26.6.28. Appl., 31.8.21. Ger., 8.3.18. Renewed 21.9.27).—See G.P. 347,672; B., 1922, 548 A.

Melting glass etc. R. L. FRINK (B.P. 291,883, 11.3.27).

Manufacture of sheet glass. R. L. FRINK (B.P. 292,448, 11.3.27).

IX.—BUILDING MATERIALS.

Application of Grosny paraffin mazout, mixed with creosote, to the preservation of wood. A. VORONOV (Neftjanoe Chozjajstvo, 1927, 12, 550—553; Chem. Zentr., 1927, II, 1526).—When creosote and petroleum products are mixed, resinous precipitates are often formed which inhibit the entrance of the preservative into the wood. Less precipitate is formed the lighter is the creosote (which must boil below 350°) and the heavier and more free from resin the petroleum product. A mixture of equal parts of creosote and paraffin mazout impregnated the wood satisfactorily.

W. S. NORRIS.

PATENTS.

Manufacture of waterproof plastic Portland cement. E. C. R. MARKS. FROM INTERNAT. PRECIPITATION CO. (B.P. 291,988, 15.8.27).—Portland cement or clinker (10—20 pts.) is ground with 1 pt. of a waterproofing and plasticising agent consisting of shale and/or diatomaceous earth containing not less than 5% and preferably 10—50% of oil or bituminous material in a form which can be extracted with solvents. When the proportion of soluble oil is less than 10%, oil is added to the shale in such quantity that the total soluble oil exceeds 10%. Other substances, *e.g.*, magnesium, calcium, or zinc chloride, sulphate, silicate, or fluosilicate, etc., may also be added. L. A. COLES.

Preparation of cement. ELEKTROSCHMELZE G.M.B.H. (G.P. 436,052, 25.6.25).—Aluminous material is melted together with a highly conducting substance in an electric furnace.

A. R. POWELL.

Hardening and ageing calcined gypsum products. J. H. COLTON, ASSR. to PACIFIC PORTLAND CEMENT CO., CONSOL. (U.S.P. 1,659,971, 21.2.28. Appl., 7.9.26).—Casting plaster is produced by calcining calcium sulphate at the required temperature, adding raw gypsum to the product, and further calcining the mixture to reduce its water content to 4—6%.

H. ROYAL-DAWSON.

Manufacture of bricks, tiles, etc. H. and T. PARDOE and H. HILL (B.P. 291,960, 14.6.27).—Mixtures of slate debris or colliery shale with ferric oxide ores or siliceous iron oxide ores, finely ground with the addition of water, if necessary, until they bind together when squeezed in the hand, are pressed or moulded into shape, dried, and baked.

L. A. COLES.

Production of acid-proof structures. J. K. WIRTH (B.P. 292,334, 26.5.27).—Acid-proof bricks are cemented together and metal surfaces etc., after application of an intermediate acid-proof layer, are coated with unhardened phenol-aldehyde condensation products, and these are subsequently hardened by treatment with acids or acid salts at the ordinary or at a slightly

raised temperature. Alternatively, uncondensed phenol-aldehyde mixtures are used and condensation and hardening are effected in one operation after application.

L. A. COLES.

Manufacture of marble substitutes. K. STICHA, J. HUBÁČEK, and J. KNEIFL (B.P. 292,388, 5.9.27).—A glazing composition of sodium sulphate, calcium chloride, black and red colouring matters, water-glass, ammonium chloride, and water is applied at about -2° to a smoothly-ground backing, and is then dried and polished.

W. G. CAREY.

Manufacture of a porous building material. J. A. ERIKSSON (G.P. 447,194, 12.6.24. Swed., 17.3.24).—A dry mixture of hydraulic material (burnt shale or the like), slaked lime, and a metal powder which evolves gas in contact with water is ground, mixed with cement and water, and formed into shapes.

A. R. POWELL.

Preservation of wood. L. P. CURTIN (B.P. 291,857, 7.3.27).—Wood is protected from attack by wood-rotting fungi, e.g., *Fomes annosus*, by impregnation with barium hydroxide solution, or an emulsion of it with petroleum oil, the hydroxide being rapidly converted into the carbonate by the carbon dioxide in the air. Sodium carbonate solution may be used when it is not likely to be leached out of the wood.

L. A. COLES.

Production of a continuous floor for linoleum, trioline, etc. L. FRIESER and O. SEEWALD (U.S.P. 1,659,867, 21.2.28. Appl., 23.7.25. Ger., 24.10.24).—To peat or peat flour previously desiccated is added coal tar to render it waterproof, and the product hardened by treatment with magnesium chloride and magnesite.

H. ROYAL-DAWSON.

Agitators [for cement slurry]. R. E. MINOGUE (B.P. 292,714, 2.4.27).

Bitumen emulsions (B.P. 275,928).—See II. Rotary kiln (U.S.P. 1,673,051).—See X.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Alloys of iron [steels] with a low percentage of titanium. H. MATHESIUS (Stahl u. Eisen, 1928, 48, 853—858).—Addition of a small percentage of aluminothermic ferrotitanium to mild steels in the ladle immediately before pouring removes practically the whole of the sulphur content, increases the yield point to about 80% of the tensile strength, instead of 50—60%, prevents segregation, produces an even structure, and generally toughens the steel. A steel with 0.15% C and 0.23% Ti had an average tensile strength of 63 kg./mm.², an elongation of 15%, and a reduction of area of 42%. A hard-drawn wire of this steel 0.5 mm. in diam. had a tensile strength of 115 kg./mm.² A. R. POWELL.

[Copper-chromium] constructional steel. E. H. SCHULZ (Stahl u. Eisen, 1928, 48, 849—853).—A steel containing 0.5—0.8% Cu, about 0.4% Cr, 0.15% C, 0.25% Si, and 0.8% Mn has all the valuable properties, for constructional purposes, of a 1% silicon steel, but its preparation is unattended by the difficulties met with in melting and casting the silicon alloy. The tensile strength is 53—58 kg./mm.², yield point 37—43 kg./mm.², elongation 22—24%, and reduction in area 47—57%.

The new steel is readily welded and is more highly resistant to corrosion than plain carbon steel containing a small percentage of copper.

A. R. POWELL.

X-Ray investigation of the structure of quenched steels. N. J. SELJAKOV, G. V. KURDUMOV, and N. T. GOODRZOV (Rev. Mét., 1928, 25, 99—104, 222—230).—Carbon steels containing 0.84, 1.02, and 1.18% C were quenched from different temperatures, and their crystal structures, before and after tempering at increasing temperatures, were determined by the Debye-Scherrer method. The quenched steels gave diffraction patterns corresponding to (1) a body-centred tetragonal structure with nearly equal axes, and (2) some unchanged γ -iron (austenite). The parameter of the body-centred tetragonal structure was almost constant at 2.85 Å., and the ratio of the axes a/c varied from 1.0 to 1.06, increasing with the carbon content and with the temperature of quenching. The body-centred tetragonal structure is thus very similar to the body-centred cubic structure of α -iron, and is looked on as a distorted form of the latter in which the carbon atoms have not combined to form cementite; the mechanism of the change from γ - to α -iron with carbon atoms occupying interstices in the iron lattice is discussed in detail on this basis. The quenched steels give diffused diffraction lines owing to the smallness of the micro-crystals, the presence of internal strains, and to heterogeneous effects due to the irregular distribution of the carbon atoms, and to the fact that the speed of quenching is not the same throughout the specimen.

W. HUME-ROTHERY.

Critical points and the martensitic tempering of nickel and nickel-chromium castings. L. GUILLET, GALIBOURG, and BALLAY (Compt. rend., 1928, 187, 14—16).—Martensitic (or secondary) tempering trials made with castings containing 3.2—3.4% C (total), 0.9% P, and varying amounts of other elements, by heating to above the transformation point and cooling rapidly, showed that silicon raises the transformation temperature, and therefore increases the critical rate of tempering, but diminishes the tendency both for primary and secondary tempering. Manganese (0.20—0.65%), nickel, and chromium have the reverse effect on the secondary tempering, whilst the tendency for primary tempering is unaffected by manganese and diminished by nickel. Such castings may be classed as (a) easily workable after moulding and hardened by cooling in air after heating to 900°, and (b) hardened after pouring but easily workable if reheated for a short period at 650°, and subsequently hardened by heating to 900° and cooling in air.

J. GRANT.

Reduction of oxide ores. B. BOGITCH (Rev. Mét., 1928, 25, 247—261).—The reduction of oxide ores of copper, nickel, cobalt, and iron by means of carbon monoxide, wood charcoal, and coal has been investigated. The degree of reduction is independent of the quantity of reducing agent present, if the temperature is high enough for the slag to separate. Rapid fusion of oxide ores of copper, nickel, and cobalt generally prevents equilibrium between the ore and the reducing agent from being reached. The addition of fluxes increases the velocity of the reaction. Their effect is specific, and depends also on the composition of the ore. The

optimum proportion of flux can be determined only by experiment, a method for which is described.

C. W. GIBBY.

Copper-antimony alloys. NEUMARK.—See XI.
Protective coatings for metals or alloys. GARDNER.—See XIII.

PATENTS.

Rotary kiln for burning, roasting, and sintering of mineral materials. F. LUTHER, Asst. to G. POLYSIUS (U.S.P. 1,673,951, 12.6.28. Appl., 4.3.27. Ger., 9.9.26).—The material falls from the kiln into a cooling drum which has a hood over its delivery end. Air is injected into the drum in such a manner that air is also sucked into it, the amount of which can be controlled.

F. G. CLARKE.

Metallurgical furnace. E. H. BUNCE and G. T. MAHLER, Assrs. to NEW JERSEY ZINC CO. (U.S.P. 1,674,947, 26.6.28. Appl., 10.12.25).—Spaced resistances having spiral convolutions providing a circuitous path for the passage of electric current extend into a molten bath contained in the furnace chamber.

J. S. G. THOMAS.

Regenerative open-hearth furnace. S. J. CORT, T. BURNS, R. S. A. DOUGHERTY, and C. E. LEHR, Assrs. to BETHLEHEM STEEL CO. (U.S.P. 1,671,100, 29.5.28. Appl., 21.12.23).—The furnace is provided with a damper-controlled throat, the lining of which is either made of heat-resistant material or provided with water-cooling means, and the dampers to which work in slots adapted to reduce leakage of gases from the furnace.

A. R. POWELL.

Annealing furnaces. BRIT. FURNACES, LTD., and E. W. SMITH (B.P. 291,909, 23.3.27).—A furnace for annealing coils of wire.

A. R. POWELL.

Annealing of metal. H. W. BROWNSDON and KYNOCH, LTD. (B.P. 292,690, 25.3.27).—Loss of heat from hot annealed metal sheets or strips is reduced by bringing the hot metal from the furnace into close contact with cold sheet about to enter the furnace chamber, the heat interchange being effected mostly by direct conduction.

C. A. KING.

Prevention of oxidation during the annealing of metal [iron or steel] bands or wires. O. H. DÖHLNER (G.P. 447,143, 21.4.26).—The material to be annealed is passed between two movable plates of refractory material during its passage through the muffle.

A. R. POWELL.

Reduction of [iron] ores. H. G. C. FAIRWEATHER. From NYBERGS GRUFABRIK AB (B.P. 291,822, 31.1.27).—Iron ore contained in carriages is passed through a long channel furnace divided into three zones, in the first of which it is subjected to a high temperature in an oxidising atmosphere to remove sulphur and roast the ore, in the second it is partially reduced by a current of reducing gases, and in the third it is completely reduced by means of carbon monoxide, which is continually regenerated by the circulation of the gas from this zone through a mass of glowing carbonaceous material. The excess gas produced in the continuous reduction process is used to effect the preliminary reduction in the second zone, and the gases from this zone, together with preheated air obtained from the cooling

chambers, are used for providing the heat necessary for roasting the ore. Water is used for quenching the partially air-cooled product from the furnace, and the steam generated passes to the recarbonising furnace for the production of water-gas, which may be used in the reduction zone.

A. R. POWELL.

Rust-proofing of iron, steel, and other metals. W. H. COLE (B.P. 292,666, 19.3.27).—Metal articles are heated in a revolving container in contact with a dry mixture of emery, copper-zinc precipitate, tin, and aluminium, to which may be added cobaltous chloride, ferrous chloride, nickel sulphate, antimony trichloride, etc.

C. A. KING.

Hardening of iron or steel articles. DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT, FORM. ROESSLER (B.P. 277,030, 5.9.27. Ger., 6.9.26).—A carburising bath for hardening purposes comprises a mixture of 40% of alkali cyanide with sodium carbonate and chloride together with 3–97% of carbon, preferably wood or animal charcoal, or with substances, e.g., anthracene or naphthalene, which liberate carbon at high temperatures. Alternatively, a mixture of 85% of sodium cyanide, 12% of sodium cyanamide, and 3% of finely-ground wood charcoal may be used. The baths may be operated at temperatures up to 950° without frothing.

A. R. POWELL.

Production of hard, wear-resisting printing surfaces. K. W. SCHWARTZ, Assr. to UNITED CHROMIUM, INC. (U.S.P. 1,673,779, 12.6.28. Appl., 5.8.25).—An unhardened steel body bearing the design to be printed has a coating of electrolytic chromium on its printing surface.

H. ROYAL-DAWSON.

Etching of [aluminium] printing plates. F. GUTZSCHKE (U.S.P. 1,660,366, 28.2.28. Appl., 17.2.27. Ter., 22.2.26).—A dilute solution of calcium nitrate and potassium dihydrogen phosphate is claimed.

H. ROYAL-DAWSON.

Ascertaining the degree of absolute or relative resistance to rusting shown by iron, steel, or iron alloys. C. MAULER (B.P. 292,794, 28.7.27).—Specimens of iron or steel alloys, particularly rustless steel, are immersed in a boiling solution containing 60–70 kg. of sodium hydroxide and 8–15 kg. of sodium nitrate in 20–40 litres of water, a little of the used solution also being added. As the solution evaporates and the b.p. rises, the specimens are withdrawn and examined with respect to the degree of rusting, which is classified in relation to the b.p. of the solution at which rusting commences. Alkalinity of the solution is maintained by adding caustic lime at intervals.

C. A. KING.

Treatment of manganese steel. V. C. MEKEEL, Assr. to TAYLOR-WHARTON IRON & STEEL CO. (U.S.P. 1,671,870, 29.5.28. Appl., 27.2.26).—In order to impart greater strength and wear-resisting properties to manganese steel without impairing its toughness or ductility the metal is case-hardened, during the regular heat-treatment, in a closed vessel packed with barium carbonate and charcoal or with boron trioxide and a reducing agent, and subsequently quenched out of contact with the air.

A. R. POWELL.

Raising the yield point of steel alloys. F. KRUPP A.-G. (B.P. 282,015, 21.10.27. Ger., 7.12.26).—A hollow

rotary body of nickel-manganese steel is rotated in a centrifugal pit at a high speed so that it undergoes a stress exceeding its yield point. The deformed rotary body is then given its final shape and will have a yield point beyond that required in service.

A. R. POWELL.

Concentration of oxidised ores. T. S. CARNAHAN, Assr. to UNION MINIERE DU HAUT-KATANGA (U.S.P. 1,671,698, 29.5.28. Appl., 23.5.27; Belg., 28.5.26).—A warmed mixture of partly rancid palm oil (1–4 pts.) and oleic acid (1 pt.) gives a better and more efficient foam than oleic acid alone when added to the ore pulp.

C. HOLLINS.

Froth-flotation concentration of ores. C. H. KELLER, Assr. to MINERALS SEPARATION NORTH AMER. CORP. (U.S.P. 1,671,590, 29.5.28. Appl., 24.3.27).—One of the usual frothing agents is used in conjunction with an impure metal xanthate, *e.g.*, the calcium xanthate obtained from lime, carbon disulphide, water, and alcohol. The process is especially useful for obtaining copper, lead, and zinc concentrates low in iron.

C. HOLLINS.

Preparation of flotation concentrates. H. J. STEHLI (U.S.P. 1,673,891, 19.6.28. Appl., 30.7.26).—In preparing plastic ore fines for sintering, a mass of wet fines is divided into lumps, and these are coated with powdered material to prevent them from adhering to one another and to allow for air passages in the mass.

H. ROYAL-DAWSON.

Roasting and/or sintering of fine [zinc] ore or other fine material. COMP. DES MÉTAUX OVERPELT-LOMMEL (B.P. 284,248, 9.3.27. Belg., 26.1.27).—The ore is roasted until the sulphur content is reduced to 5–6% of sulphide sulphur. The product is mixed with 1–5% of ferrous sulphate or sulphuric acid and with sufficient water to form a plastic mass. This mass is fed into a machine which compresses it and extrudes it as friable cylindrical lumps which readily break up into coarse sand amenable to blast roasting or sintering in the Dwight-Lloyd or Schlippenbach apparatus.

A. R. POWELL.

Smelting of zinc ores. H. SEIDLER (G.P. 445,689, 16.1.25).—The roasted ore is smelted with a deficiency of carbon so that only about half of the zinc is recovered as metal. The sintered residue in the retorts is freed from any powdery carbonaceous material it contains, mixed with an excess of fuel or reducing agent, and smelted in blast or revolving furnaces to obtain zinc oxide for use as a pigment, and to recover any other easily reducible metal contained in the residue.

A. R. POWELL.

Bath for galvanising. N. K. TURNBULL (B.P. 291,961, 14.6.27).—In internally-heated galvanising baths (*cf.* B.P. 220,398; B., 1924, 836) the rate of transfer of heat from the layer of lead to that of zinc is increased by means of metallic conducting bars establishing thermal connexion between the layers and passing through the interposed layer of dross.

J. S. G. THOMAS.

Treatment of pyritic [tin] ores, residues, etc. F. L. WILDER, E. MORRIS, E. SCHIFF, and E. S. KING (B.P. 291,919, 6.4.27).—The roasted material is ground to a fine sand which is leached under pressure in an

autoclave successively with an ammoniacal solution of ammonium carbonate, an alkaline brine, and an acid brine. The first treatment removes copper and tungsten, the second stannic acid, and the third lead, bismuth, and silver, whilst the residue contains cassiterite and the precious metals.

A. R. POWELL.

Dearsenification of ores and metallurgical products. NORDDEUTS. AFFINERIE (B.P. 286,285, 17.10.27. Ger., 3.3.27).—Arsenic is removed from arsenide ores or speiss by heating a mixture of the finely-ground arsenical material with 25% of pyrites and 20% of fine bituminous coal in a multiple-hearth furnace to which a restricted supply of air is admitted. The charge is self-combustible, and if mechanically rabbled does not cake.

A. R. POWELL.

Treatment of oxidised arsenical lead ores. DR. A. NATHANSOHN, METALL- U. FARBWERKE A.-G., OTAVI MINEN- U. EISENBAHN-GES., A. HIRSCH & SOHN, ZINK-HÜTTE HAMBURG, and COMP. MÉT. FRANCO-BELGE DE MONTAGNE (SOC. ANON.) (G.P. 445,854, 1.11.24).—Arsenical lead ores are leached with a concentrated solution of a chloride containing free hydrochloric acid, and the arsenic dissolved is precipitated by the addition of lime followed by oxidising substances with or without the addition of a ferric salt.

A. R. POWELL.

Extraction of metals from metallic sulphides. **Extraction of metallic nickel.** A. B. BAGHDASARIAN [A. B. BAGSAR] (U.S.P. 1,671,003—4, 22.5.28. Appl., [A] 17.8.25, [B] 19.7.26).—(A) The sulphides are chloridised at a suitable temperature, and the chlorides so produced are heated and reduced with hydrogen to give the metals. (B) A solution containing nickel is first treated to remove objectionable impurities, and then most of the nickel is precipitated by addition of metallic zinc. This is collected, the clear solution is boiled, and to it is added more zinc to complete the precipitation of the nickel.

F. G. CROSSE.

Cyaniding apparatus for treating gold ores. R. KANDA (U.S.P. 1,673,982, 19.6.28. Appl., 13.9.26. Japan, 24.9.25).—The cyaniding tank has a conical bottom to which is connected an annular pipe through which inlet nozzles project at a tangent into the tank. These nozzles communicate with the annular pipe from which they convey liquid to the tank.

A. R. POWELL.

Treatment of ores. W. E. STOKES, Assr. to U.S. PROCESSES, INC. (U.S.P. 1,674,806, 26.6.28. Appl., 18.11.22).—Ores are roasted, at a temperature below their fusion point, with an alkali bicarbonate.

F. G. CROSSE.

Workable copper-silicon alloys resistant to corrosion. F. DOEBLIN (G.P. 447,247, 11.7.24).—Copper alloys resistant to corrosion by alkalis and acids contain 5–10% Si, 1.5–12% Ni, CO, and/or Fe, and up to 5% Cr, W, Mo, Ti, or V.

A. R. POWELL.

Manufacture of [nickel] alloy. R. FRANKS and B. E. FIELD, Assrs. to HAYNES STELLITE CO. (U.S.P. 1,675,798, 3.7.28. Appl., 8.1.25).—The alloy contains 3–5% Al, 3.5–6% Si, 5–12% W, 6–12% Zr, up to 1% B, and the rest chiefly nickel.

F. G. CROSSE.

Treatment of nickel-copper-aluminium alloys. T. S. FULLER, Assr. to GEN. ELECTRIC CO. (U.S.P.

1,675,264, 26.6.28. Appl., 17.3.24).—The physical properties of such alloys containing only a small proportion of aluminium are improved by deforming the alloys at about 1000° and completing the treatment at 500—1000°.

F. G. CROSSE.

Bimetallic element. H. SCOTT, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,671,490—1, 29.5.28. Appl., 27.12.26).—A thermostatic element comprises strips of two elements having different coefficients of expansion, the element having the lower coefficient being (A) a ferrous alloy containing 38—45% Ni or (B) an alloy of 45% Ni and 55% Fe, and the element having the higher coefficient being (A) iron with 10% Mn and 10% Ni or (B) a steel alloy containing 0.1% C and 15—23% Mn. The elements are very susceptible to temperature changes between 150° and 400°.

A. R. POWELL.

Soldering lead-bearing metal castings. METALLBANK U. METALLURGISCHE GES. A.-G. (G.P. 447,133, 23.1.25).—The molten bearing metal is treated with a small quantity of magnesium and poured into its bed which has previously been coated with a suitable solder.

A. R. POWELL.

[Aluminium] alloy. Heat-resisting alloy and structure. N. V. HYBINETTE (U.S.P. 1,675,708—9, 3.7.28. Appl., [A] 23.9.22. Renewed 17.11.27. [B] 2.2.24).—(A) An alloy contains at least 90% Al, substantial amounts of nickel and iron, considerably less than 1% of chromium, tungsten, or molybdenum, and traces only of copper. (B) The alloy contains 5—10% Ni, 15—25% Cr, 1—2% C, 0.5—2% Al, and remainder iron.

F. G. CROSSE.

Welding medium for aluminium. W. REUSS (U.S.P. 1,675,664, 3.7.28. Appl., 3.12.27. Ger., 24.7.25).—A mixture of equal parts of borax, boric acid, and caustic potash is claimed.

F. G. CROSSE.

Treatment of metals. [Coating wires with aluminium.] G. H. HOWE, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,673,624, 12.6.28. Appl., 2.1.25).—Metal wire is passed downwards into a bath of molten aluminium, then upwards through the bath in a direction parallel to its downward path. At the lowest point of the bath the wire operates a baffle, which sets up a circulation in the bath, and the metal is heated by means of a gas flame at the point at which the wire leaves it.

A. R. POWELL.

Treatment of aluminium alloys. H. C. HALL and T. F. BRADBURY (B.P. 292,407, 5.11.27. Addn. to B.P. 281,912; B., 1928, 128).—The casting previously described is brought to the required degree of hardness and tensile strength by being heated at 160—190° for at least 20 hrs., and then quenched.

H. ROYAL-DAWSON.

Removal of oxide films from metal surfaces. H. WAGNER (G.P. 446,652, 3.5.25. Addn. to G.P. 368,537).—The surface is covered with a mixture of alkali, lime, zinc dust, and a polyhydric phenol, xanthic acid, or thiocarbamide, and the metal heated until deoxidation is effected.

A. R. POWELL.

Drying and removing gases from metal powders. E. C. R. MARKS. From RINGSDORFF-WERKE A.-G. (B.P. 291,949, 28.5.27).—Moist metal powders are

slightly compressed and alcohol is forced, sucked, or centrifuged through the mass to displace water, whereupon the mass is allowed to dry in the air either with or without being compressed into any desired shape.

A. R. POWELL.

Working-up of mixed shavings of white metal and red metal. A. SCHWARZ, Assee. of INTERNAT. METALL A.-G. (B.P. 270,740, 5.5.27. Ger., 5.5.26).—To separate the constituents of mixed shavings obtained in the turning of bearings, the mixture is heated to the m.p. of the eutectic of the white metal in a rotating drum provided with baffles, whereby the white metal is broken up into small particles by the abrasive action of the red metal (bronze) turnings. Subsequent sieving of the mixture effects almost complete separation of the powdered white metal from the unchanged red metal.

A. R. POWELL.

Apparatus for the electrolytic production of metallic sheets and similar articles. J. C. F. A. SCHÜTTE (B.P. 292,015, 14.11.27).—An apparatus for the recovery of sheet metal by electrolysis direct from ore or scrap metal comprises a wood or stoneware vat, the walls of which are lined with lead which serves as the cathode. The anode consists of lump ore or scrap metal packed in the annular space between two perforated cylinders each of which can be rotated independently or together in the same or in opposite directions. A rotating paddle device within the inner cylinder further assists in the free circulation of the electrolyte throughout the vat.

A. R. POWELL.

Cupola. A. H. COPLAN, Assr. to ECONOMY METAL PRODUCTS CORP. (U.S.P. 1,675,255, 26.6.28. Appl., 10.6.27).—See B.P. 288,072; B., 1928, 412.

Manufacture of ferroboration and boron-steel. T. MIYAGUCHI (U.S.P. 1,674,119, 19.6.28. Appl., 19.10.21).—See B.P. 195,678; B., 1923, 661 A.

Manufacture of copper alloys. O. VON ROSTHORN (U.S.P. 1,675,008, 26.6.28. Appl., 11.5.21. Ger., 4.2.20). See B.P. 158,882; B., 1922, 596 A.

Production of technical pure beryllium respectively for freeing metallic beryllium from impurities. H. FISCHER, Assr. to SIEMENS & HALSKE A.-G. (U.S.P. 1,673,043, 12.6.28. Appl., 7.12.25. Ger., 10.9.25).—See G.P. 443,944; B., 1928, 58.

Recovery, separation, or extraction of gold, platinum, and other metals, applicable also for separation of precious stones from earth or material containing them. G. C. E. F. HANGIAU (U.S.P. 1,673,675, 12.6.28. Appl., 25.9.23. U.K., 9.10.22).—See B.P. 213,626; B., 1924, 522.

Treatment of ores containing refractory oxide. T. R. HAGLUND, Assr. to INTERNAT. PATENT CORP. (Re-issue 17,001, 19.6.28, of U.S.P. 1,569,483, 12.1.26).—See B., 1926, 164.

Drying and extracting gases from metallic powders. E. SEYFFERTH, Assr. to FULMIT GES.M.B.H. (U.S.P. 1,674,230, 19.6.28. Appl., 16.6.27. Ger., 12.3.25).—See B.P. 291,949; preceding.

Treatment of ores (B.P. 279,447).—See II. **Electron-emission material** (U.S.P. 1,670,463).—See XI.

XI.—ELECTROTECHNICS.

Addition agents in electrodeposition. V. Application of the complex cation theory of crystalloidal addition agents to base metals. I. G. FUSEYA and K. MURATA. II. G. FUSEYA and R. YUMOTO (*J. Soc. Chem. Ind. Japan*, 1928, 31, 323—331, 331—342; cf. B., 1926, 950).—I. In an attempt to show the formation of complex cations between glycine present as an addition agent and lead or zinc ions, by spectroscopic and electrometric methods, it was found by the former method that a mixed solution of lead fluosilicate or perchlorate and glycine extended its absorption bands toward longer wave-lengths in the ultra-violet zone than did its components, thus indicating the formation of complex compounds in both solutions. With a mixture of zinc sulphate and glycine no such increased absorption occurred. Similarly, ferric and chromic sulphates formed complex compounds with glycine, but none was detected spectroscopically in the case of nickel and cobalt sulphates. From measurements of the *E.M.F.* of cells of the type: $\text{Pb}|\text{0.1N-Pb}(\text{NO}_3)_2|\text{0.1N-Pb}(\text{NO}_3)_2 + M\text{-glycine}|\text{Pb}$, and the degree of ionisation of salt solutions calculated from conductivity ratios, the degree of complex formation was computed. It was found that about 94% of lead and about 75% of zinc were in complex forms in their 0.1N-solutions when 1 mol. of glycine was present, and these compounds by migration experiments were found to be cations.

II. No increase in weight of the deposits nor decrease in size of the crystals was found in the electrodeposition of lead and zinc from lead nitrate, lead fluosilicate, and zinc sulphate solutions containing glycine. It is concluded that in such electrolytes the glycine does not behave as an addition agent but forms complex cations with these metallic ions.

S. OKA.

Anodic behaviour of copper-antimony alloys. H. NEUMARK (*Metall u. Erz*, 1927, 24, 305—311; *Chem. Zentr.*, 1927, II, 1391—1395).—Electrolysis of alloys of antimony and copper in sulphate electrolytes with or without the addition of tartaric acid soon ceases owing to the formation of a non-conducting film of basic salts of copper and antimony on the anode. Copper may, however, be recovered from antimonial alloys by electrolysis in a solution of copper nitrate. No antimony enters the electrolyte and the current efficiency is high. The anode slime consisting chiefly of antimonious acid does not adhere to the anode, so that the electrolysis can be carried to completion. If the alloy contains lead the best results are obtained with a slightly acid bath which is 2N with respect both to copper sulphate and copper nitrate.

A. R. POWELL.

Duriron as an insoluble anode for gold cyanide solutions. M. DE K. THOMPSON [with COSGROVE, SARROS, HAUEISEN, and DINAN] (*Amer. Electrochem. Soc.*, Sept., 1928, 54. Advance copy. 5 pp.).—Corrosion experiments show that duriron anodes are much more resistant than peroxidised lead anodes in cyanide solutions of the concentration used in the electrolytic extraction of gold. Duriron anodes cannot be used, however, for the oxidation of chlorate to perchlorate, of chromic salts to chromate, or of manganate to permanganate.

H. J. T. ELLINGHAM.

Speed controller. VEIHMAYER and GIVAN.—See XVI. **Purification of water.** ATEN.—See XXIII.

PATENTS.

Electric annealing furnace. SIEMENS-SCHUCKERTWERKE G.M.B.H., Assees. of T. STASSINET (B.P. 262,800, 9.12.26. Ger., 12.12.25).—In an electric annealing furnace for intermittent working comprising a heating chamber formed by fireclay walls carrying resistances and surrounded by insulating walls, the fireclay wall is of minimum thickness consistent with strength and resistance, whilst the thickness of the insulating walls is such that the sum of the accumulator heat loss and that due to radiation and conduction is a minimum.

J. S. G. THOMAS.

Optical pyrometer. D. C. GALL (B.P. 291,971, 29.6.27).—In an optical pyrometer of the disappearing-filament type, in which the voltage drop in the filament is taken as the indicator of temperature, potential leads are connected to parts of the filament which are not appreciably affected by changes in the temperature of the surroundings. When the resistance of the filament inserted in a form of Wheatstone bridge is employed as temperature indicator, a compensating filament resistance inserted in one arm of the bridge eliminates errors due to the temperature of parts of the principal filament, e.g., the ends, being affected by changes in the temperature of its surroundings or by conduction etc.

J. S. G. THOMAS.

Magnetic core material. STANDARD TELEPHONE & CABLES, LTD. From WESTERN ELECTRIC CO., INC. (B.P. 290,736, 19.2.27).—An iron-nickel alloy containing more than 25% Ni is treated with less than 5% of antimony, and the molten mixture is thoroughly oxidised by means of air, oxygen, or a metal oxide. The product is broken up, the fragments are coated with an insulating material, and the product is made into loading coils. (Cf. U.S.P. 1,669,649; B., 1928, 490.)

A. R. POWELL.

Apparatus and method for gaseous reactions. J. M. WEED, Assr. to GEN. ELECTRIC CO. (U.S.P. 1,673,654, 12.6.28. Appl., 23.4.24).—Gases are supplied under pressure to the region outside the porous, refractory wall of a chamber in which an arc is operated, and means are provided for carrying away gases which have passed into the arc through pores in the chamber wall.

J. S. G. THOMAS.

Incandescence electric lamp. R. A. PRICE, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,673,267, 12.6.28. Appl., 15.5.24).—The lamp filament, supported by insulating material, is positioned by an alloy containing more than 50% Au and more than 10% Ag.

J. S. G. THOMAS.

Filaments for electric incandescence lamps. SOC. FRANÇ. DES LAMPES À INCANDESCENCE "LUXOR," Assees. of J. A. M. HAWADIER (B.P. 281,685, 1.12.27. Fr., 1.12.26).—A metallic core of relatively high m.p., e.g., of tungsten, is covered with a metal of lower m.p., e.g., molybdenum, and this is coated with a material, e.g., chalk, magnesium carbonate, which has great illuminating power at a temperature below the m.p. of the metallic coating.

J. S. G. THOMAS.

[Electron-emission] composition. H. L. BERGER (U.S.P. 1,671,007, 22.5.28. Appl., 19.1.27).—A composite material of high emissive power is produced by boiling silver in an alkaline-earth hydrogen sulphide solution. A. R. POWELL.

Preparation of electron-emission material [thorium-molybdenum alloy]. J. W. MARDEN, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,670,463, 22.5.28. Appl., 11.4.23).—A mixture of molybdenum powder and the compound Th_3Al_4 is formed into bars, which are sintered in a high-frequency induction furnace *in vacuo* until the aluminium is volatilised and the thorium is thoroughly incorporated with the molybdenum. The alloy is then swaged, forged, and drawn or rolled as required. Alternatively, a molybdenum plate or anode is coated with Th_3Al_4 and heated in an evacuated valve whereby the thorium becomes incorporated with the molybdenum and the aluminium acts as a getter in the valve. A. R. POWELL.

Activation of electron-emission material. T. P. THOMAS, Assr. to WESTINGHOUSE LAMP CO. (U.S.P. 1,670,487, 22.5.28. Appl., 14.4.23).—The material is coated with thorium oxalate, introduced into an evacuated vessel, and heated to decompose the oxalate and liberate thorium. F. G. CROSSE.

Arc lamp electrodes. H. BECK (G.P. 446,881, 17.5.24).—The electrodes are provided with an outer layer of chromium which either volatilises completely or forms a porous slag. A. R. POWELL.

Primary cell. M. L. MARTUS and E. H. BECKER (U.S.P. 1,673,198, 12.6.28. Appl., 5.10.26).—A self-depolarising cell has an electrolyte of caustic alkali, and a negative element of activated carbon extending above the normal level of the electrolyte.

H. ROYAL-DAWSON.

[Diatomaceous absorbing medium for] battery. E. J. PETRIE and G. R. HANNAN (U.S.P. 1,673,421, 12.6.28. Appl., 10.3.26).—Electrolyte is absorbed in a medium consisting of diatomaceous shells substantially freed from foreign matter by heating to a temperature above the drying temperature and sufficiently high to drive off volatile constituents and destroy organic matter, but which is below the temperature at which fusion of the diatoms occurs. J. S. G. THOMAS.

Utilisation of the contents of used dry batteries. F. TROSTLER, and CHEM. FABR. JOHANNISTHAL G.M.B.H. (G.P. 447,740, 23.7.26).—The material is dried and intimately mixed with sulphur. The mixture is burnt in a multiple-hearth furnace with indirect firing, and the product is treated in an oil flotation machine. The concentrates are extracted with acid to remove manganese compounds and obtain a carbon of higher electrical conductivity than the original graphite used in making the batteries. A. R. POWELL.

Lead plates for accumulators. E. F. DRAISMA (B.P. 291,833, 22.2.27).—A plate of soft lead is enclosed in a frame or support of a lead-antimony alloy.

J. S. G. THOMAS.

Regeneration of the metal content of electrolytic [chromium] baths. ELEKRO-CHROM-GES.M.B.H. (G.P. 446,249, 28.12.23).—An auxiliary chromium cathode is

short-circuited with the anode of the plating bath, and the current strength of the auxiliary circuit is regulated by an electromagnet operated by the current in the main circuit, preferably through a relay. A. R. POWELL.

Electrolytic condenser and electrolyte therefor. H. N. MILLER, Assr. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,672,899, 12.6.28. Appl., 5.3.27).—Filmed aluminium and conducting plates are retained in spaced relation by a dry gelatin-glycerin composition containing sodium bicarbonate. J. S. G. THOMAS.

Electrolytic condenser. E. F. ANDREWS, Assr. to ANDREWS-HAMMOND CORP. (U.S.P. 1,673,434, 12.6.28. Appl., 7.11.27).—A positive electrode composed of a nickel-iron alloy is used. J. S. G. THOMAS.

[Dry] battery cell. H. CSANYI, Assr. to L. KLOPMAN and W. W. HUSELTON (U.S.P. 1,675,973, 3.7.28. Appl., 17.9.24).—See B.P. 240,140; B., 1925, 997.

[Electrical] protection of metallic parts against corrosion. F. VON WURSTENBERGER (U.S.P. 1,671,173, 29.5.28. Appl., 30.4.24. Switz., 14.5.23).

Bitumen for insulation (B.P. 291,858).—See II. Metallurgical furnace (U.S.P. 1,674,947). Metallic sheets (B.P. 292,015).—See X. Articles from synthetic resins (B.P. 291,477).—See XIII. Electrodeposition of rubber (B.P. 291,471).—See XIV.

XII.—FATS; OILS; WAXES.

Composition of the fatty acids present as glycerides in Elasmobranch oils. T. P. HILDITCH and A. HOULBROOKE (Analyst, 1928, 53, 246—257).—The oils examined consisted of (a) the mixed residual oils (after removal of squalene by vacuum distillation) from the livers of various species, including *Scymnorhinus lichia*, *Centrophorus granulosus*, *Lepidorhinus squamosus*, and *Etmopterus spinax*, and (b) the residual liver oil from *Scymnorhinus lichia*. Methods used were broadly those described previously (B., 1928, 306), and in the case of the first-named residue a check of the main results was obtained by analysing the fatty acids after complete hydrogenation. The total combined fatty acids from (a) contained solid acids 61.2%, liquid acids 38.8%. The solid and liquid acids, respectively, consisted of myristic 0.8, 0.2; palmitic 11.8, 1.4; stearic 1.2, 0.1; arachidic 1.2, —; myristoleic —, 0.2; palmitoleic 1.1, 2.4; oleic 18.2, 17.2; gadoleic 9.0, 7.4; cetoleic 12.3, 3.5; selacholeic 5.6, 6.4%. The composition of (b) agreed fairly closely with the above up to the C_{18} acids, but there was a difference of 6% less of oleic acid, and more cetoleic acid was present. Compared with normal marine animal oils, the degree of unsaturation of the fatty acids is relatively low, and they contain but little less saturated than monoethylenic derivatives, but the non-fatty matter is exceedingly highly unsaturated. Although C_{20} and C_{22} acids are present in about the same proportion as in other marine animal oils, they are mainly monoethylenic; there is also about 10–15% of a monoethylenic C_{24} acid present. D. G. HEWER.

Tung oil. D. HOLDE, W. BLEYBERG, and M. A. AZIZ (Farben-Ztg., 1928, 33, 2480—2484).—The iodine value (Hanus) of tung oil has been found to rise markedly

with increasing time of reaction from $\frac{1}{4}$ hr. to 2 hrs., and afterwards to remain steady at 230–240, corresponding to saturation of the three double linkings of the elæostearic acid present. A 0.2*N*-solution of iodine bromide in carbon tetrachloride gave a similar result in 3 hrs. As with other vegetable oils a constant iodine value is obtained after $\frac{1}{4}$ hr., it is suggested that the presence of tung oil in linseed oil might be recognised quantitatively by observation of an iodine value increasing with the time of reaction, and *vice versa*. In the case of β -elæostearic acid, the iodine value rose from 176 to 279 during $\frac{1}{2}$ –2 hrs.' treatment (45% excess of iodine bromide); 60% excess of the reagent gave iodine values up to 300, and, in a few cases, values up to 350 (equivalent to four double linkings) were attained, resulting possibly from the occurrence of substitution or of traces of water in the glacial acetic acid etc. Solutions of iodine or bromine alone were less reactive than was iodine bromide. E. LEWKOWITSCH.

Organic peroxides as bleaching agents: application to oils and fats. F. V. HOORT (Oil & Fat Ind., 1928, 5, 180–182).—The use of benzoyl peroxide as an oxidising bleaching agent for oils and fats is suggested. The reagent (about 0.5–0.2%) is mixed to a paste with some of the oil, and the mixture added to the bulk, which is heated to 80–90°. The whole is stirred and left for a sufficient time (20 min. or longer). The most suitable proportion of reagent, temperature and time of reaction should be determined by preliminary laboratory tests for each oil to be treated. No filtering is necessary, and steaming removes residual benzoic acid. E. LEWKOWITSCH.

Report of Du Boscq Colorimeter Committee. D. WESSON (Oil & Fat Ind., 1928, 5, 173–175).—To test the suggestion that by the use of the Du Boscq colorimeter it should be possible to read the colour of cottonseed oils in terms of "prime summer yellow," duplicate sets of samples were tested by four different laboratories. Considerable differences in the readings were shown (greater than with the Lovibond tintometer). Matching was complicated by differences in hue due to the presence of several different colouring matters, and to the effect of bleaching etc. of the oils, and different results were obtained with different depths of the standard oil. It is concluded that the Du Boscq instrument, although suitable for measuring relative amounts of the same colouring matter in optically similar liquids, needs modification for successful application to oil colorimetry. E. LEWKOWITSCH.

Paraffin waxes. MEYERS and STEGEMAN.—See II.

PATENTS.

Manufacture of acid- and lime-resisting derivatives of unsaturated fatty acids. I. G. FARBENIND. A.-G. (B.P. 288,127, 23.12.27. Ger., 1.4.27).—A lower fatty acid or its anhydride is treated with fuming sulphuric acid to give the corresponding acyl derivative, which is then coupled with an unsaturated hydroxy-fatty acid, anhydride, or ester. The process has the advantage that it may be carried out at moderate temperatures and gives light-coloured products of good stability. E. HOLMES.

Production of sulphonation products from fats, oils, or their acids. H. T. BÖHME A.-G., and H. BERTSCH (B.P. 274,104, 8.7.27. Ger., 8.7.26).—Fatty materials which may be sulphonated by means of sulphuric acid in the presence of anhydrous strong organic acids, or their anhydrides or chlorides, give products of enhanced utility if they are first polymerised. Thus "Floricin," which results from the dry heating of castor oil, may be used (cf. B.P. 261,385; B., 1928, 418). E. HOLMES.

Apparatus for sulphonating oils. A. HORWITZ (U.S.P. 1,671,586, 29.5.28. Appl., 22.6.21).—Uniform sulphonation of large batches of oil is achieved by forcing the sulphonating acid under air pressure through numerous nozzles placed above the surface of the oil, which is stirred in an open tank fitted with cooling coil and run-off valve. C. HOLLINS.

Purification or separation of mixtures of fatty or mineral oils. J. Y. JOHNSON. From I. G. FARBENIND A.-G. (B.P. 291,817, 7.1.27).—The application of solvents consisting of the formate of a lower fatty alcohol or of mixtures of such formates is claimed. Thus methyl formate readily removes free acids from sesamum oil, sulphur compounds from Mexican crude oil, and a golden-yellow resin from the higher oily residue from the catalytic hydrogenation of a crude rock oil. E. HOLMES.

Wax-like compounds (B.P. 291,849, 292,056, 292,058, and 292,057).—See III. **Fatty still residues.** (U.S.P. 1,669,490–1).—See XIV.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Loss of material of paints by weathering. H. A. MÖLLER (Farben-Ztg., 1928, 33, 2484–2486).—Quantitative experiments on the weathering of white lead paints spread on sheet iron were made and the "half-life periods" of the coatings calculated for comparison. Grey-white lead paints proved notably more resistant than white, the weathering varying according to the nature of the black pigment incorporated. Sulphate white lead paints preserved the surface gloss longer, collected less dirt, and had a longer life than those containing carbonate white lead. In all cases loss of material was less for those paints having a red lead undercoat. E. LEWKOWITSCH.

Composition of zinc chrome. B. A. ELLIS, J. J. FOX, and J. F. HIRST (J. Oil and Colour Chem. Assoc., 1928, 11, 194–198).—The divergence in composition assigned to zinc chrome in the literature is due to incomplete methods of analysis and to different methods of manufacture. A complete scheme of analysis is detailed and the results of examination of commercial zinc chromes of various types are tabulated. The probable composition $5\text{ZnO} \cdot 4\text{CrO}_3 \cdot \text{K}_2\text{O} \cdot 4\text{H}_2\text{O}$ is indicated for potassium zinc chrome and $2\text{ZnO} \cdot 2\text{CrO}_3 \cdot (\text{NH}_4)_2\text{O} \cdot \text{H}_2\text{O}$ for the dark-orange ammonium zinc chrome, but approximate agreements with stoichiometrical ratios are not considered of much importance for such complicated substances. S. S. WOOLF.

Precipitation of zinc chromate. J. L. BUCHAN and J. J. FOX (J. Oil and Colour Chem. Assoc., 1928, 11, 198–200).—A description of some experimental precipitations of zinc chromes, undertaken in order

to ascertain how the various types of commercial pigments (cf. preceding abstract) may be obtained.

S. S. WOOLF.

Titanium dioxide rubber enamels. R. DITMAR (Chem.-Ztg., 1928, 52, 506—507).—Fine-grained titanium dioxide is very suitable for the production of snow-white and highly luminous, coloured rubber enamels, since it has a better white-colouring power than, for example, zinc oxide, improves the rubber material, and, unlike colouring agents containing zinc, is non-poisonous. Such enamels may be produced from vulcanised rubber since the latter is rendered very plastic and adhesive by the titanium oxide. By the use of sulphur monochloride vapour the enamels may be vulcanised in 6 min. instead of the usual 34—44, giving well-aged and more durable products.

B. P. RIDGE.

Malayan damars. II. T. H. BARRY (J. Oil and Colour Chem. Assoc., 1928, 11, 203—216; cf. B., 1925, 815).—An account of the characteristics, composition, and analytical constants of dead damar, black damar, and damar Minyak.

S. S. WOOLF.

[Liebermann]-Storch-Morawski reaction for rosin. E. ELSNER (Farben-Ztg., 1928, 33, 2547—2548).—The unreliability of the positive Liebermann-Storch-Morawski reaction for rosin is discussed. It is further shown that solutions of rosin and various rosin products in turpentine give a negative reaction up to a concentration of 10%, and a very fleeting positive reaction at somewhat higher concentrations. A negative rosin test obtained on varnishes containing turpentine cannot, therefore, be relied on, contrary to previous views.

S. S. WOOLF.

Resins of lignite. WIGGINTON.—See II.

PATENTS.

Preparation for stripping paint, varnish, and the like. F. C. DEAN and O. P. SWIFT (B.P. 291,812, 8.12.26).—A preparation which remains wet is made from pure benzol, crude benzol (or coal-tar naphtha), methylated spirit, paraffin wax, and ceresin wax.

B. FULLMAN.

Production of printing ink. J. J. JAKOSKY (U.S.P. 1,673,245, 12.6.28. Appl., 1.11.26).—A portion of a quantity of oil suitable for use as a vehicle in printing ink is heated locally to a temperature sufficiently high to decompose it partly into carbon and gaseous products, when it is immediately mixed with the surrounding oil. The process is continued until at least part of the oil contains sufficient carbon for use as printing ink, and this is then separated from the remaining oil.

L. A. COLES.

Manufacture of water-colour printing ink. J. HOEFFLER, Assr. to J. HOEFFLER & Co., Inc. (U.S.P. 1,660,196, 21.2.28. Appl., 12.3.27).—A mixture of gum arabic solution and an excess of glycerin is dehydrated by heat to produce a paste, a pigment is added, and the product ground.

H. ROYAL-DAWSON.

Manufacture of coloured solutions of nitrocellulose. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 291,539, 10.3.27).—Insoluble mineral or organic colouring matter in water or other indifferent liquid is mixed (in a Kollergang or kneading machine etc.) with

nitrocellulose wet with water, in the absence of gelatinising agents, to homogeneity. The product is pressed, freed from water by extraction or heat, and dissolved in the usual varnish solvents.

B. FULLMAN.

Coating materials. [Water japans.] BRIT. THOMSON-HOUSTON CO., LTD., Assees. of W. P. DAVEY (B.P. 275,955, 25.7.27. U.S., 16.8.26).—To the water japan made according to B.P. 212,248 (B., 1925, 557) ammonia is added. The coatings electrically deposited from the japan are thickened and improved.

B. FULLMAN.

Coating by depositing emulsified japan ingredients. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of H. CHISLET (B.P. 275,960, 26.7.27. U.S., 16.8.26).—Surfaces are coated with a solution of an electrolyte (preferably an alkali), dried, and dipped into an aqueous japan emulsion. The disperse phase in a layer of the emulsion is coagulated by the electrolyte, forming an adherent layer on the surface.

B. FULLMAN.

Production of varnish bases and masses insoluble in water. G. WALTER (B.P. 262,148, 29.11.26. Austria, 23.11.25).—The primary condensation products from formaldehyde and carbamide or thiocarbamide are heated rapidly to above the m.p., with the addition of carbamide, thiocarbamide, or acid amides in general, and/or alcohols (benzyl alcohol), together with acids, such as chloroacetic or acetic acid with or without buffer salts. The additions may be made to the solid or molten condensation product, and, if desired, a non-aqueous solvent (volatile or not) may be employed. Glassy or white masses of varying degrees of hardness are obtained according to the ingredients and conditions. The use of much acetamide and benzyl alcohol leads, after addition of volatile solvent (methyl alcohol), to clear lacquers. The products may be hardened by a further heat treatment; the process and the hardening may be performed under pressure.

C. HOLLINS.

Treatment of rosin. R. C. PALMER, Assr. to NEWPORT CO. (U.S.P. 1,660,762, 28.2.28. Appl., 4.10.24).—A mixture of zinc chloride and rosin is heated at 250—330°, and the product distilled *in vacuo* to produce rosin oils.

H. ROYAL-DAWSON.

Resinous reaction product of urea [carbamide] and formaldehyde. F. LAUTER, Assr. to ROHM & HAAS CO. (U.S.P. 1,671,596, 29.5.28. Appl., 7.5.26).—In order to avoid cloudiness due to methylenecarbamide, hot carbamide is added to a boiling solution of formaldehyde.

C. HOLLINS.

Manufacture of articles from synthetic resins or shellac. DUNLOP RUBBER CO., LTD., and R. C. DAVIES (B.P. 291,477, 3.12.26).—Synthetic resins, *e.g.*, of the phenol-formaldehyde type, or mixtures of these with natural resins, *e.g.*, shellac or rubber, in the form of aqueous dispersions are electrodeposited on an anode or on a porous mould surrounding the anode. Instead of using preformed resins, the dispersion may contain the resins in a partially formed condition or the components for forming the resins. Compounding ingredients also may be present in the dispersion. The deposit, which may be left on the anodic mould or may be stripped therefrom, is then dried and heated to complete the hardening.

D. F. TWISS.

Manufacture of plastic masses from albuminous substances, like casein. R. and M. SCHRODER, E. JAROSLAW, and S. LEVIS [JAROSLAW'S ERSTE GLIMMER-WAREN-FABR. IN BERLIN] (B.P. 272,947, 17.6.27. Ger., 17.6.26).—Alkaline solutions of albuminous substances, *e.g.*, casein, blood, intestinal mucus, etc., containing a phenol are treated with an aldehyde in the presence of ammonia. B. FULLMAN.

Hardening of plastic masses from casein and the like. I. G. FARBERIND. A.-G. (B.P. 268,804, 31.3.27. Ger., 31.3.26).—Hardening takes place during hot pressing if the plastic mass contains compounds (*e.g.*, methylolureas and urethanes) which, in the temperature range employed, yield formaldehyde and difficultly volatile substances which improve the properties. B. FULLMAN.

Hardening resinous exudations. E. A. DANIELS and H. S. SNELL, ASSTS. to WESTERN ELECTRIC Co., INC. (U.S.P. 1,673,803—8, 19.6.28. Appl., 21.8.24).—Resinous exudations are condensed with (A) a guanidine, (B) aldehyde-ammonia, (C) an aminonaphthalene, (D) a secondary aromatic amine, (E) a piperidine-carbon disulphide additive product, (F) sodium β -naphthoxide. L. A. COLES.

Condensation of urea and formaldehyde under pressure. A. GAMS and G. WIDMER, ASSTS. to Soc. CHEM. IND. IN BASLE (U.S.P. 1,674,199, 19.6.28. Appl., 4.1.26. Switz., 17.1.25).—See B.P. 246,127; B., 1927, 684.

Mills for reducing paints, colours, and pigments. J. R. TORRANCE, and TORRANCE & SONS, LTD. (B.P. 292,431, 3.1.28. Addn. to B.P. 209,829; B., 1924, 238).

Blanc fixe (G.P. 445,668). Lakes from phosphotungstomolybdenum compounds (B.P. 292,253).—See VII. Acid-proof structures (B.P. 292,334).—See IX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Ageing of vulcanised rubber. IV. Relation between wave-length of light and deterioration of vulcanised rubber. T. YAMAZAKI (J. Soc. Chem. Ind. Japan, 1927, 30, 804—813).—Vulcanised rubber became darker when merely heated in the dark at 70°, but when exposed to sunlight at 60—70° the change in colour was far more marked. The higher the degree of vulcanisation, the greater was the colour change. Moreover the rubber was hardened by the action of light, such action being quicker when the wave-length of the light was shorter and the temperature higher. The action of light of 4300Å. was feeble, except for over-vulcanised rubber, which was noticeably hardened even by light of longer wave-length. Acetone extract increased with time of exposure, the rate of increase being less when light of longer wave-length was used. In some cases the acetone extract reached a maximum and then decreased. Remarkable decrease in free sulphur by the action of light and heat was also noticed. Y. NAGAI.

Ageing of vulcanised rubber. V. Effect of sunlight and heat on the mechanical properties of vulcanised rubber. VI. Effect of sunlight filtered through coloured glass on mechanical properties of vulcanised rubber. T. YAMAZAKI (J. Soc. Chem.

Ind. Japan, 1928, 31, 233—242, 243—257).—The time of vulcanisation of rubber-sulphur mixes was found to have a marked effect on the ageing properties. The deterioration due to sunlight was great even in winter, and still more pronounced in spring at 60°. The content of acetone-soluble matter increased during ageing and the content of free sulphur decreased. The outer layers of test pieces exposed to sunlight were quite brittle and non-elastic, but the inner layers were fairly good. Test pieces subjected to Geer tests showed little difference between outer and inner layers. Test pieces exposed to the light through a coloured glass filter transparent down to 3300Å. showed serious deterioration even at 40°, but those exposed to a filter transparent to 4360Å. showed only slow deterioration at 40° but a somewhat marked one at 65°. S. OKA.

Homogeneous rubber and the conception of an elastic molecule. H. FEUCHTER (Kautschuk, 1928, 4, 103—106, 118—123).—Features in common between the characteristics of liquid crystals and those of the elastic-plastic state of rubber are indicated, together with the possibility of applying knowledge of the former to elucidation of the constitution of rubber. The conversion of anisotropic racked rubber into the isotropic material probably represents, in principle, the same thermal function as the melting of liquid crystals. The rubber fibres are regarded as bundles or parallel groupings, possibly spiral in configuration, of units with a linear molecular character, which presumably represent the chains of isoprene or dimethyloctadiene nuclei suggested earlier, *e.g.*, by Pickles, Harries, or Staudinger. From the possibility of removing the racking effect, it is necessary to postulate the existence of periodic weaker linkings between some of the isoprene links in a chain; the linking arrangement in rubber, therefore, resembles in some respects an elastic lattice or cross-woven fabric. Rubber in its various elastic forms can be regarded as a dynamic, completely reversible mixture of an ideal elastic, or "iso-elastic," rubber ("ideale Kautschuk"), to which "diffusion rubber" is an approximation, with another ideal hard, vitreous form ("glasartige Kautschuk"), which is designated "para-elastic."

D. F. TWISS.

Rubber enamels. DITMAR.—See XIII.

PATENTS.

Production of rubber [by electrodeposition]. S. O. COWPER-COLES (B.P. 291,471, 20.11.26, 14, 15, 16, and 22.12.26, 8.1, 3.2, 11.2, 14.6, and 2.8.27).—In the production of rubber sheets, tubes, etc. from latex, containing compounding ingredients if desired, by electrodeposition on an anode mould of cadmium or zinc, the escape of anodic bubbles is facilitated and homogeneity of the deposit is ensured by effecting deposition intermittently, *e.g.*, by an intermittent or alternating current, by periodic withdrawal of the anode from the latex, or by rotating the anode on a horizontal axis giving only partial immersion of the mould. A soft, electrically-conductive deposit is ensured by neutralisation of the ammonia in preserved latex with acetic acid. Metal powder may be incorporated in the deposit by dusting or by electrodeposition subsequent to the formation of the rubber deposit. Electrodeposition of rubber is also

effected by spraying latex from a negatively-charged nozzle on to an anodic mould. D. F. TWISS.

Treatment of latex and products obtained therefrom. W. A. GIBBONS and J. MCGAVACK, ASSTS. to U.S. RUBBER PLANTATIONS INC. (U.S.P. 1,673,672, 12.6.28. Appl., 11.5.27).—Latex containing an anti-coagulant which permits bacterial action is kept until the protein constituents are substantially decomposed. On evaporation a transparent film is obtained with a pitted surface. D. F. TWISS.

Manufacture of rubber articles. M. C. TEAGUE, ASSR. to AMER. RUBBER CO. (U.S.P. 1,673,649, 12.6.28. Appl., 25.7.25).—A relatively thick layer of an aqueous dispersion of rubber, in the presence of a hydrophilic agent, is exposed to drying conditions, whereby moisture is withdrawn substantially uniformly from all parts without the formation of a dried surface film.

D. F. TWISS.

Manufacture of hollow rubber articles. DUNLOP RUBBER CO., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 291,479, 4.12.26).—Hollow articles of rubber or similar material are formed from a concentrated and/or compounded dispersion on a temporary form or mandrel, e.g., of fusible metal, wax, or clay, which may subsequently be removed as a liquid or as a suspension in a fluid. Vulcanisation can be effected before or after removal of the form. D. F. TWISS.

Production of rubber-like bodies. W. B. PRATT, ASSR. to DISPERSIONS PROCESS, INC. (U.S.P. 1,671,314, 29.5.28. Appl., 17.2.26).—Natural rubber is imitated by dispersing a terpene (isoprene, dipentene, or polymerides) in water in presence of a hydrophilic protective colloid of protein character, the terpene particles being about the size of the particles in rubber latex. The coating of protective colloid is then tanned by the addition of cresol, creosole, or other tanning agent, so as to prevent rupture of the globules during milling. When coagulated the product forms a rubber-like mass capable of vulcanisation, swelling in organic solvents without rupture of the coatings. Natural or synthetic resin may be added to give an acetone-soluble ingredient.

C. HOLLINS.

Reclaiming and dispersing vulcanised rubber. T. G. RICHARDS and G. P. F. SMITH, ASSRS. to DISPERSIONS PROCESS, INC. (U.S.P. 1,671,316, 29.5.28. Appl., 17.5.27).—In the alkali process for reclaiming vulcanised scrap rubber, a saponifiable substance (resin oil, acid resins, pine tar, higher fatty acids) is added so that a soap is formed *in situ*, which enables the reclaim to be dispersed readily in water.

C. HOLLINS.

Vulcanisation accelerator. H. O. CHUTE (U.S.P. 1,673,801, 19.6.28. Appl., 21.12.22).—An accelerator is prepared by adding carbon disulphide to a mixture of magnesia with an aromatic amine in the cold, and subsequently heating below 140° until evolution of hydrogen sulphide is practically complete.

L. A. COLES.

Vulcanised pitches. Treatment of fatty still residues. W. SAVAGE, ASSR. to SAVAGE-RUBBER CORP. (U.S.P. 1,669,490—1, 15.5.28. Appl., [A] 17.1.22, and [B] 12.6.23).—The black tarry residue obtained as a by-product in the refining of fatty acids by distillation is

vulcanised (A) with 7% of sulphur at 150—205° in the presence of zinc oxide and thiocarbanilide, or (B) with more than 5% of sulphur at 135—205° in the presence of an organic accelerator base and an active alkaline compound, e.g., magnesia, magnesium carbonate, or lead oxide. The products are soft, rubbery substances, which may be hardened by the introduction of any of the usual rubber fillers.

A. R. POWELL.

Covering of wire, thread-like and filamentary material, etc. with india-rubber, gutta-percha, balata, etc. DUNLOP RUBBER CO., LTD., D. F. TWISS, and E. A. MURPHY (B.P. 291,485, 28.1.27).—Wire and thread-like material is passed through a bath of concentrated and/or compounded aqueous dispersion of rubber, gutta-percha, or balata, so that it issues therefrom with a uniform coating of the dispersion. In leaving the bath the filament conveniently passes through a short constricted tube one end of which is immersed in the bath. The coating is dried without contact with any foreign material. The filament may consist of colloidal material capable of being absorbed into the coating during subsequent drying or vulcanisation.

D. F. TWISS.

Antimony pentasulphide (U.S.P. 1,671,203).—See VII.

XV.—LEATHER; GLUE.

Depilation [of skins] by mould enzymes. G. ABT (J. Soc. Leather Trades' Chem., 1927, 11, 520—530).—*Aspergillus oryzae*, *A. effusus*, *A. parasiticus*, *A. ochraceus*, and *A. flavus* contain enzymes capable of loosening the hair on skins. The most active is *A. oryzae*, which produces an amylolytic enzyme, a lipase, and a protease. It is the protease which causes depilation, but the lipase probably assists. It is most active at a much lower temperature than pancreatin. It will loosen the hair at 20°, but 25—28° is the optimum temperature and 35—38° is best for goatskins. 0.4% boric acid solution or 0.4—0.8% sodium bicarbonate solution should be added to the unhairing liquor as an antiseptic. For depilation the process occupies about 24 hrs. Dried skins should be soaked back before treatment, and goatskins should be treated in water at 28°. The action of the mould enzymes is stimulated by pretreating the skins with a 0.2—0.4% solution of sodium hydroxide for 24 hrs. The optimum effect of the mould protease on casein solution is at p_H 6.5—7.0, but the optimum unhairing effect appears to be at p_H 8.0. The two effects are not the same. Sections of skins unhaird by this mould show the entire removal of the epidermis, the hair bulbs, fat, and sweat glands, and good separation of the fibrils. The appearance resembles that obtained after the best bating materials. Pancreatin appears to attack the elastic and collagen fibres, whilst the protease in *A. oryzae* has a selective action on the epidermal tissues, the epidermal mucous layer, and the hair structures. It also exerts much less hydrolytic action on collagen than does pancreatin, but it hydrolyses limed collagen fairly readily.

D. WOODROFFE.

Chamoising of skins. P. CHAMBARD and L. MICHALLET (J. Soc. Leather Trades' Chem., 1927, 11, 559—576).—Chamoising consists of two different processes: the tannage proper, in which the collagen is converted

into a new non-hydrolysable substance with changed properties, and a filling or adsorption process, in which a certain amount of saturated glycerides lubricate the leather. Various theories as to the reactions taking place during chamoising have been critically examined, and experimental work has failed to confirm either the peroxide or the aldehyde theory, to the exclusion of the other. It is concluded that the intermediate oil-oxidation products are chiefly responsible for the tannage, and that the secondary adsorption process is essential to the properties of chamois leather. The products of complete oxidation and of polymerisation of the neutral glycerides and fatty acids are absorbed by the skin. The oxidation and polymerisation products of the oil are soft, clear, and amorphous, whilst the polymerised fatty acids are brownish-black, hard, and brittle. Hence the difference between skins tanned with fish oils and fatty acids respectively. Neutral cod oil is the best chamoising agent and produces a leather of good quality, but the tannage is slow. Free fatty acids furnish a rapid method of tannage, but produce a harsh leather. The most practicable chamoising oils contain some free acids. Resinates of cobalt, iron, lead, and manganese catalyse the chamoising in order of decreasing effect. 0.1% can be used to increase the amount of oil combined with the pelt after 24 hrs.

D. WOODROFFE.

Tanning barks in Madagascar. F. H. DE BALSAC and A. DEFORGE (J. Soc. Leather Trades' Chem., 1927, 11, 551—558).—Analyses of five samples of mimosa bark, *Acacia decurrens*, showed water 9.39—12.65%, tans 24.3—27.84%, soluble non-tans 5.36—10.24%, insolubles 51.86—57.2%, and ratio of tan/non-tan 2.7—5.3. Their tannin content was lower than that of Natal mimosa bark, but the ratio of tan/non-tan was equal to or better than that of the Natal bark. The Madagascar barks are not suitable for export, but only for local use or the manufacture of dry extract. Other barks examined are: badamier (*Terminalia Catappa*), lalona (*Weinmannia Bojeriana*, Tul), filao (*Casuarina equisetifolia*, Forst), rotra (*Eugenia sp.*), fany (*Peptadenia chrysostachys*, Benth), and sakoa (*Solerocarya caffra*, Sonder), the percentage tans and non-tans in which are: 11.8, 4.4; 13.7, 2.7; 15.3, 3.7; 12.6, 12.2; 3.1, 17.0; and 3.5, 4.2, respectively. Fany, sakoa, and rotra contain too much non-tans to be useful in tanning. Lalona gives too dark a tannage for European tanners, and if used for extract manufacture it would require decolorising. Filao is the most promising material for cultivation purposes. D. WOODROFFE.

Chromium collagenates. C. SCHIAPARELLI and G. BUSSINO (J. Soc. Leather Trades' Chem., 1927, 11, 531—550).—Samples of delimed bated pelt were shaken with 3% and 5% solutions of chrome alum respectively. The increase in weight and the temperature of gelatinisation (T_g) were noted at regular intervals. T_g increased as the weight increased. The rate of tannage and rise in T_g decreased at a certain stage and then increased again. This was probably due to the conversion of chromium collagenate into dichromium collagenate. The tanned pelt contained 3.91% Cr_2O_3 at this stage, compared with 3.38% for monochromium collagenate, and had T_g 92—94°. The concentration

of the chrome alum solution did not affect the nature of the tannage, but increased its speed. Tannage with a 5% solution of basic chrome alum (basicity 16.6%) showed a break at T_g 108—110° (Cr_2O_3 8.41%) which is attributed to the conversion of dichromium collagenate (Cr_2O_3 6.76%) into the trichromium compound. Tannage with a liquor of basicity 33.3% showed a break at T_g 120° (in glycerol) and the pelt contained on the average 9.37% Cr_2O_3 . With a liquor of 50% basicity, the T_g increased to 130°, tetrachromium and a little pentachromium collagenate being formed. T_g was constant at 134—138° with a liquor of 66.6% basicity, and the increase in weight was nearly double that obtained with the liquor of 50% basicity. A heptachromium collagenate has been obtained, but not the octachromium compound (cf. Thomas and Kelly, B., 1922, 640 A). The results confirm Wilson's theory on chromium collagenates and the preponderating chemical nature of chrome tannage with chrome alum. Ageing is the conversion of simple chromium collagenates into higher compounds, with the elimination of acid. A 16.6% basic chrome alum solution suffices for a light chrome tannage, and a 33.3% solution is most suitable for "box" or moderate tannages. Experiments show that the boiling test does not indicate complete tannage. The determination of T_g would afford a better control.

D. WOODROFFE.

Synthetic tannins. L. MEUNIER and C. CASTELLU (J. Soc. Leather Trades' Chem., 1927, 11, 495—507).—See B., 1928, 133.

Determination of insoluble [matter] in tanning extracts. A. TURNBULL (J. Soc. Leather Trades' Chem., 1928, 12, 202—3).—See B., 1928, 534.

PATENTS.

Manufacture of tanning agents. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 291,245, 18.6.27).—The water-soluble products obtained from phenols (phenol, *m*-cresol, *p*-chlorophenol) by condensation with formaldehyde and a sulphite are heated with sulphur and water in presence of alkali, particularly the alkali liberated when a neutral sulphite is used with the formaldehyde. C. HOLLINS.

Production of [metal]-decorated articles from casein. A. I. G. WARREN, and PRECIOUS METAL INDUSTRIES, LTD. (B.P. 291,877, 10.3.27).

XVI.—AGRICULTURE.

Soils of Prince Edward Island. F. T. SHUTT (Can. Dept. Agric., 1928, Bull. No. 100, 20 pp.).—Analyses of a large number of cultivated soils are recorded. For the maintenance of fertility, the adoption of green manuring supplemented by the judicious use of fertilisers and lime is recommended.

A. G. POLLARD.

Exchange acidity in primary soils. R. PÜRCKHAUER (Z. Pflanz. Düng., 1928, 11A, 359—386).—The weathering of primitive rocks tends to produce an acid soil which becomes more neutral during cultivation. Examination of numerous soil profiles indicates that the reaction of the primary soil lies within a fairly narrow range of p_H values and is not appreciably influenced by the depth of the weathered stratum or the nature of the

original rock. Forest growths tend to intensify the acidity of these soils. Differences in the reaction of meadow and arable land are small and traceable to differences in manuring. Pasturage maintains a favourable reaction, and use for garden cultivation tends to produce an alkaline reaction. Acid fertilisers do not markedly influence the p_H value of this type of soil, but lime causes profound changes. Natural manures are the most suitable as the soils usually have low buffer capacities. Good growth of clover is possible with soils of p_H value less than 4.5. In acid soils white charlock (*Raphanus raphanistrum*) occurs in its deep yellow modification, and a relationship is suggested between this phenomenon and the p_H value of the nutrient medium.

A. G. POLLARD.

Nature of base exchange and soil acidity. H. W. KERR (J. Amer. Soc. Agron., 1928, 20, 309—335).—The exchange reactions of soils are ascribed to the open structure or colloidal nature of the complex, and a mass-action equation for the heterogeneous equilibrium is formulated. Zeolitic materials appear to be present in the soil. Experiments with colloidal aluminosilicic acid show that acids need not be in true solution in order to give acid p_H values by an electrometric method. The difference in the chemical behaviour of colloidal and dissolved materials is of degree rather than of kind.

CHEMICAL ABSTRACTS.

Replaceable bases and dispersion of soil in mechanical analysis. M. D. THOMAS (Soil Sci., 1928, 25, 419—427).—The maximum amount of dispersion is to be obtained in the majority of cases by producing sodium saturation in the soil mass. In view of the experimental difficulty attending the preparation of sodium-saturated soil it is likely that a treatment with dilute acid followed by the addition of sodium carbonate is the best routine method of dispersing non-calcareous soil.

H. J. G. HINES.

Aqueous vapour pressure of soils. IV. Influence of replaceable bases. M. D. THOMAS (Soil Sci., 1928, 25, 484—493; cf. B., 1928, 538).—The vapour pressure-moisture curves of soils treated to exchange their bases for a single base are greatly influenced by the nature of the replaceable base. In dry soils the potassium-treated material has the least and the calcium-treated material the greatest water-absorbing power, but at high moisture contents the sodium-saturated material has the greatest absorbing power. In the study of a colloidal mineral it was shown that various hydrates of its sodium complex exist. Soils exhibit similar properties to this mineral, and it is suggested that the soil colloidal material resembles it in composition.

H. J. G. HINES.

Studies with sulphur for improving alkali soil. J. D. HAYNES (Soil Sci., 1928, 25, 443—446).—The permeability of the soil was improved (a) by alternate wetting and drying, (b) by treatment with sulphur. Lime and manure maintained this improvement. Of the substances examined, sulphur was the most effective in decreasing the p_H of the soil.

H. J. G. HINES.

Behaviour of the nitrogen of spent wash in the soil. E. PARISI (Annali Chim. Appl., 1928, 18, 198—204).—The absorptive power of soil for the amino-

acids of spent wash from molasses distilleries is either feeble or non-existent. The ammonification of the amino-acids and betaine occurs regularly in the soil, but is very slow, so that there is danger of the washing-out of these substances. Losses due to the development of gaseous nitrogen in soils fertilised by spent wash and containing a proper proportion of moisture are practically negligible, and even when large amounts of the slop are used, the nitrogen is transformed almost quantitatively into nitrates.

T. H. POPE.

Calcium carbonate-soil equilibrium and the lime requirement. S. MATTSO (Soil Sci., 1928, 25, 429—431).—When calcium carbonate is added to the soil, part of the calcium is adsorbed and part dissolved as bicarbonate by the carbon dioxide so liberated. By placing soil colloidal material in a parchment bag which was in contact with a paste of calcium carbonate, it was shown that the soil-calcium carbonate equilibrium was reached in seven days. By replacing the adsorbed calcium with ammonium chloride, the degree of unsaturation, and hence the lime requirement, can be found.

H. J. G. HINES.

Rate of availability of various forms of sulphur fertilisers. J. D. HAYNES (Soil Sci., 1928, 25, 447—453).—A sulphur preparation inoculated with *Thiobacillus oxidans*, a colloidal sulphur preparation, and other commercial by-products were mixed with soil, and the rate of oxidation to sulphate was measured. Of the by-products, black gas-sulphur (the colloidal preparation) was oxidised as rapidly as the inoculated sulphur. On alkali soils sulphur decreased the p_H value, increased the microflora of the soil, and produced a decided deflocculating effect.

H. J. G. HINES.

Do soils become more acid on drying? W. BRENNER (Z. Pflanz. Düng., 1928, 11A, 141—144).—The reaction of normal soils is but slightly altered on drying.

H. J. G. HINES.

Influence of pulverisation and drying of soil on its productivity. A. ACHROMEIKO (Z. Pflanz. Düng., 1928, 11A, 65—89).—The drying of soil in the sun results in increased solubility in water of phosphates and organic matter. Repeated drying multiplies this effect, which is even more pronounced on oven-drying or steam-sterilisation. On re-wetting the soil reversion occurs. Similar effects on the reaction were also noticed and ascribed to microbiological action. The alterations in structure on drying and re-wetting are due mainly to the changes in the organic matter.

H. J. G. HINES.

Phosphate applications and their influence on chernosem [soils]. IV. Reversion of phosphates in soils. M. A. EGOROV (Soil Sci., 1928, 25, 463—472; cf. Egorov and Mackov; B., 1926, 763).—The addition of sodium phosphate to soil causes a large part of the organic matter to become water-soluble. Sodium hydrogen phosphate gives a less coloured extract. It is proposed to use this action on the soil organic matter to follow the changes in composition of various phosphates when added to the soil.

H. J. G. HINES.

Chemical determination of soil fertility. K. BAMBERG (Z. Pflanz. Düng., 1928, 11A, 115—141).—The

phosphoric acid in soils (particularly humus soils) is rendered more soluble in citric acid by drying; the solubility in nitric acid, however, is but little changed. A comparison of the Mitscherlich and Neubauer methods with a number of chemical methods was carried out on a large number of soils. The chemical methods included extraction of phosphates and potash with citric acid, ammonium citrate, nitric acid, and mixtures of ammonium citrate and nitric acid. It is concluded that the value of chemical methods cannot be disputed, and that they do give true indications as to the response of soils to phosphatic manuring.

H. J. G. HINES.

Theoretical considerations on the determination of soil nutrients. S. GERICKE (Z. Pflanz. Düng., 1928, 11A, 144—150).—The citric acid extraction method is criticised on the ground that the final reaction of the extracted soil varies according to the initial characteristics of the soil. In the Neubauer method the influence of the added sand modifies the texture of heavy soils considerably, and hence no true notion of the actual behaviour of the soil is obtained. The seedling method also takes no account of the influence of one nutrient on the amount of another nutrient taken up by the plant.

H. J. G. HINES.

Use of the Dumas-Dennstedt method in the determination of total nitrogen in soils and agrarian products. E. PARISI (Annali Chim. Appl., 1928, 18, 196—197).—For determining nitrogen in soil, flour, and other agricultural products, Dennstedt's method is recommended in place of that of Dumas. The hard glass combustion tube may be replaced by one of opaque quartz and the manganese carbonate or sodium hydrogen carbonate used as a source of carbon dioxide by a Kipp's apparatus. If 8—10 g. of soil are taken, duplicate tests agree within about 2—3%.

T. H. POPE.

Determination of organic matter and especially humified substances in soil. U. SPRINGER (Z. Pflanz. Düng., 1928, 11A, 313—359).—Comparative examination of existing methods is recorded. Among processes for determining the total organic matter in soils a modified chromic acid method is recommended. Methods for the selective analysis of humic material by extractive processes are unsound, in that portions only of the humic matter are removed (except in the case of sodium hydroxide extracts) and also some undecomposed plant residues are frequently affected. A modification of Grandean's method (extraction with 5% caustic soda solution and oxidation with permanganate) yielded results generally satisfactory for comparative work, but lower than those obtained by the acetyl bromide method. During the colorimetric examination of alkaline extracts of soil some decomposition and loss of colour intensity may occur. Of the direct methods that of Lapicque and Barbé, whilst of little value for quantitative measurements, yields valuable information as to the state of decomposition of humus and is simpler than those of König and of Robinson and Jones. The acetyl bromide method is the most accurate of those examined.

A. G. POLLARD.

Use of highly viscous fluids in the determination of volume weight of soils. S. H. BECKETT (Soil Sci.,

1928, 25, 481—483).—A rapid and accurate determination can be made by removing the soil with an auger, and collecting and drying the sample; the cavity thus formed is filled with heavy oil or cane syrup, the container and oil being weighed before and after the operation.

H. J. G. HINES.

Simple speed controller, especially adapted to the moisture-equivalent centrifuge [for soils]. F. J. VEIHMAYER and C. V. GIVAN (Soil Sci., 1928, 25, 455—460).—Control is established in the electrically-driven machine by a vibrating reed in a shunt circuit.

H. J. G. HINES.

Determination of the toxic substance in insecticides. IV. Volumetric determination of mercury in seed preservatives. J. BODNÁR, L. E. RÖTH, and I. TERGINA (Z. anal. Chem., 1928, 74, 81—105. Cf. B., 1926, 997; 1927, 30, 856).—The destruction of the organic matter may be carried out without loss of mercury by heating the substance with sulphuric acid and nitric acid or, preferably, hydrogen peroxide in a conical flask fitted with a reflux air condenser 40 cm. long. The operation takes 30 min. with the peroxide and 1—2 hrs. with nitric acid. In either case the solution, if free from chlorides, is diluted and titrated directly with thiocyanate; if chlorides are present it is made just alkaline with sodium hydroxide and the mercury precipitated by addition of formaldehyde. After 30 min. the precipitate is collected on a close-textured paper, washed, and dissolved in nitric acid for titration; an addition of 0.6 mg. of mercury is made to allow for slight loss during destruction of the organic matter in the presence of chlorides.

A. R. POWELL.

Boll-weevil poisons. WALKER.—See VII. **Toxicities of arsenicals and fluorine compounds.** MARCOVITCH.—See XXIII.

PATENTS.

Drying or curing hay or like crops. H. WADE. From A. J. MASON (B.P. 292,211, 9.3.27).—The freshly-cut crop is formed into a matted layer of uniform thickness and density before being submitted to the drying process. Suitable apparatus is described.

A. G. POLLARD.

Fungicide. I. G. FARBENIND. A.-G. (B.P. 271,480, 18.5.27. Ger., 18.5.26).—The product contains a complex organic mercury compound soluble in alkalis, e.g., sodium mercuriolyxide, and a copper compound soluble in alkalis, e.g., a mixture of copper sulphate and sodium potassium tartrate, with or without the addition of other fungicides, wetting agents, or inert material.

L. A. COLES.

Germicides (B.P. 292,222).—See XX.

XVII.—SUGARS; STARCHES; GUMS.

Wood-sugar problem. I. Saccharification of the cellulose. P. LEONE and A. NOERA (Annali Chim. Appl., 1928, 18, 205—239).—A critical summary is given of previous work on this subject, both in the laboratory and on an industrial scale. The experimental results given show that, when hydrochloric

acid of 36–37% concentration acts on cellulose, the greatest concentration of the resulting dextrose solution is 13.66%, the amount of acid employed being about seven times that of the sugar obtained. Addition of zinc chloride to the acid raises the attainable concentration of the sugar and lowers the consumption of acid. Thus with a solution containing 37% of hydrogen chloride and 20% of zinc chloride, a 34% sugar solution is obtainable with the value 3 for the ratio acid : sugar. These results are of interest since, with 40% hydrochloric acid alone, the sugar concentration never exceeds 30%, and if sawdust is treated with such acid counter-currentwise, the mean final concentration of the sugar is 28%.
T. H. POPE.

Boiling of white sugars. F. NOSEK (Z. Zuckerind. Czechoslov., 1928, 52, 441–447).—Insoluble particles present in the liquor from which white sugars are boiled (fibres, char dust, mineral matter, etc.) if sufficiently fine may serve as grain in the subsequent crystallisation, causing the sugar to be dull. Their presence is therefore to be avoided, though the greyish tinge of granulated sugar may also be due to occluded air bubbles. In forming grain by “seeding,” the sugar dust used should be boiled from the best liquors, and care must be taken thoroughly to disperse it throughout the contents of the pan, otherwise conglomerates are formed. Dust obtained by grinding is less suitable than specially boiled very fine grain. Fine “pilé” should not be used. In boiling white sugars the formation of the grain and its growth can be followed to advantage under the microscope, and useful information is thus obtained as to the conditions requisite for the production of uniform crystals.
J. P. OGILVIE.

Use of permutite for the purification of [beet] sugar juices. I. MUTTI and C. REGINELLI (Zymologica, 1928, 3, 91–95).—The sodium and potassium present in a beet juice prepared in the laboratory may be almost completely removed by treatment with calcium permutite, which acts also as a decolorising, filtering material and retains considerable proportions of mucilaginous substances removable only with difficulty during the regenerative washing of the permutite; this difficulty may possibly be overcome by ignition of the permutite.
T. H. POPE.

Methods of decolorising sugar solutions by means of active carbons. V. SÁZAVSKÝ (Z. Zuckerind. Czechoslov., 1928, 52, 413–420).—In the suspension method a certain amount of the carbon is mixed with the juice during 15 min. at about 80° and pumped through filters of suitable type, whilst in layer filtration the carbon is previously deposited in a layer about 25 mm. thick and the liquor under decoloration pumped through this. In the latter method a better utilisation of the carbon is realised by working in two stages so that the liquors are passed first through almost exhausted carbon and subsequently through fresh material. Some factories use a combination of the suspension and layer methods. Which method is the most suitable depends mainly on the nature of the carbon and on the liquors under treatment. In general, the layer method in two stages is the most favoured for high-power carbons.
J. P. OGILVIE.

Determination of chlorine in sugar factory products. C. E. BUDLOVSKÝ (Z. Zuckerind. Czechoslov., 1928, 52, 421–430).—In the case of products soluble in water or dilute nitric acid, the acidified solution is treated with a slight excess of silver nitrate, clarified with ferric phosphate, and the precipitate centrifuged and dried without washing. It is then distilled with concentrated sulphuric acid in the presence of copper in a special apparatus, the hydrochloric acid condensed in water being titrated by Votoček's method. Insoluble products are distilled with concentrated sulphuric acid in the same apparatus, following the lines of the Vaubel-Scheuer method (B., 1906, 286), and the hydrochloric acid is determined as before. Results obtained by these two methods were consistently higher than those found by determining the chlorine in the ash of the product examined.
J. P. OGILVIE.

Molybdomanganimetric determination of lactose. G. FONTÈS and L. THIVOLLE (Lait, 1927, 7, 547–555; Chem. Zentr., 1927, II, 1314).—The cuprous oxide precipitated by the action of lactose on Fehling's solution is dissolved in a solution of molybdic acid in phosphoric acid, and the resulting blue reduced solution is titrated with permanganate.
A. R. POWELL.

Lactose from whey. BELL and others.—See XIX.

XVIII.—FERMENTATION INDUSTRIES.

Hydrogen ions in brewing processes. IV. G. HAGUES (J. Inst. Brew., 1928, 34, 362–376; cf. B., 1927, 589).—There is no relationship between the duration of fermentation and the p_H value of a fermenting wort, and the hydrogen-ion concentration is the combined result of the total acidity and the buffer substances remaining in solution. In the first stages of fermentation, when yeast reproduction is active, the total acidity remains constant or falls slightly, and the simultaneous decrease of p_H value is due to a removal of buffer substances. In the latter stages acid substances are formed, and the decrease of the p_H value slows down with the concurrent increase in the buffer content of the wort. The manner in which the buffering of a wort alters during fermentation is not influenced by the initial p_H value of the wort between the values 4 to 6. The presence of gypsum in the mashing water has very little effect on the buffering of the wort. Increasing amounts of gypsum to a maximum content of 20 grains per gallon decrease the p_H value and increase the total acidity of the wort. With a further increase of gypsum the p_H value increases. The rate of yeast multiplication is optimum at p_H values 4 and 8–8.5, whilst a minimum occurs at p_H 6.2. C. RANKEN.

Sulphites [in beer]. H. HERON (J. Inst. Brew., 1928, 34, 377–385).—The determination of sulphur dioxide in beer by the Monier-Williams method, where the sulphur dioxide distilled from the acidified beer is introduced into hydrogen peroxide, gives slightly lower results than those obtained with the method recommended by the Institute of Brewing, in which bromine water replaces the hydrogen peroxide. To a wort of gravity 1045° the malt contributes 3–8 pts. of sulphur dioxide per million, whilst hops, malt extract, and various

priming syrups also yield small amounts. Although a proportion of the sulphur dioxide is oxidised in the mash or lost during boiling, an appreciable amount becomes stabilised in the wort and appears in the beer at tracking owing to its combination with certain of the wort constituents to form hydroxysulphonic acids, which are non-volatile and resistant to oxidation. The loss during storage of sulphur dioxide, which is added to the finished beer in the form of sulphites, or as a constituent of the finings, seldom amounts to more than 25% of the quantity added, which loss occurs within the first two or three weeks. C. RANKEN.

Comparison of six different strains of the organism commonly concerned in large-scale production of butyl alcohol and acetone by the biological process. E. R. WEYER and L. F. RETTGER (*J. Bact.*, 1927, 14, 399—424).—The organism is diastatic, saccharolytic, and proteolytic; all the common, and most of the rare, sugars are sources of available carbon. Nitrogen in the form of proteins or peptones is necessary; ammonium salts and simple amino-acids are inadequate. The organism is not susceptible to the antiseptic action of butylresorcinol.

CHEMICAL ABSTRACTS.

Depilation of skins. ABT.—See XV. **Yeast fermentation.** JAMES and HUBER.—See XIX.

PATENTS.

Manufacture of yeast. J. Y. JOHNSON. From H. BÜCHER (B.P. 292,202, 19.2.27).—Yeast is seeded into a slightly acid, aerated wort containing adequate amounts of commercial molasses and a combination of yeast nutrients from which no acid is produced, or from which the acid produced is sufficient for the maintenance of the optimum acidity for yeast growth. The nutrients, which are soluble in water and do not precipitate insoluble compounds among the yeast, include carbamide, ammonium nitrate, and triammonium phosphate as the substances containing nitrogen, and, in addition, various combinations of magnesium sulphate, potassium sulphate, and tricalcium phosphate may be present. By adding fresh nutrients to the wort after complete or partial removal of the yeast and using the wort over again, as many as 12 successive crops of yeast may be grown before the repeated use of the wort inhibits reproduction. C. RANKEN.

Stabilisation of substances used as ferments or antiseptics. I. G. FARBENIND. A.-G., Assocs. of R. BEILING (G.P. 446,130, 23.3.24).—The stabiliser used is dihydroxyacetone or esters or ethers derived from it.

A. R. POWELL.

Manufacture of yeast. R. L. CORBY and W. H. F. BÜHRIG, Assrs. to FLEISCHMANN Co. (U.S.P. 1,673,735, 12.6.28. Appl., 16.8.24).—See B.P. 238,554; B., 1925, 897.

XIX.—FOODS.

Factors influencing the absorption in experimental baking [of dough]. J. MICKA and E. CHILD (*Cereal Chem.*, 1928, 5, 208—214).—The quantity of flour used in a dough has little effect on the absorption. There is a difference of about 3% between the absorption of a dough consisting of flour and water alone, and

that of a dough containing salt, sugar, lard, and yeast. In the former case the end-point is more difficult to determine. The influence of the ingredients was studied separately and collectively. Salt causes the dough to be slacker at first, becoming stiffer on being kept. In the case of sugar, the initial slackening effect continues. Lard decreases the absorption slightly; yeast increases it. High absorption is more detrimental to the quality of the bread than low, the best results being obtained with 57% absorption. In reporting percentage absorption values, retention power during fermentation and other factors which affect absorption should be considered. They should not be computed to a 13.5% or 15% moisture basis. W. J. BOYD.

Yeast fermentation in flour-water suspensions. T. R. JAMES and L. X. HUBER (*Cereal Chem.*, 1928, 5, 181—191).—A device is described by which the rate of fermentation of flour-water suspensions can be automatically recorded as indicated by the quantity of carbon dioxide evolved per min. Using this method the rate of fermentation was found to be greater for clear flours than for patent flours. The addition of salt increases fermentation even at a concentration of 20 g. of salt in 500 c.c. of water and 200 g. of flour. Oxidising agents have no beneficial effect. Ammonium chloride tends to inhibit fermentation at first, but towards the end of the fermentation period the growth of the yeast is greatly stimulated. Alkalinity decreases fermentation and acidity increases it, but the effect of acidity varies greatly with the type of flour.

W. J. BOYD.

Lipoid phosphorus of wheat and its distribution. B. SULLIVAN and C. NEAR (*Cereal Chem.*, 1928, 5, 163—168).—The phosphorus content of the lipoids (*i.e.*, the alcohol-ether extract) decreases with decreasing refinement of the flours, whilst the percentage of lipoids and the total phosphorus in the flours increase. The highest grade of flour has the lowest total nitrogen content, but contains more nitrogen in its lipid than do the lower grades. The lipid material in the middlings, bran, and germ contain in proportion more neutral fats and other ether-soluble substances than the flours, and the nitrogen and phosphorus contents of the lipoids are correspondingly low. Extraction with ether alone is inadequate for the determination of phosphatides in wheat. W. J. BOYD.

Action of phosphatides in bread dough. E. B. WORKING (*Cereal Chem.*, 1928, 5, 223—234).—When dough is fermented the acid produced swells the gluten strands by increasing their water absorption and so reduces their tensile strength. At the same time the dough is rendered more ductile through lubrication of the strands by substances such as the phosphatides. These effects are necessary to produce the required loaf-volume and texture. The fermentation period can be omitted if acid and either egg-yolk or an oxidising agent be added. The oxidising agent liberates the phosphatide present in the flour. The addition of alcohol, glycerol, or saponin has a similar effect to that of egg-yolk. A baking formula is given which provides for the addition of lactic acid and sodium chlorate; this has been successfully used in a commercial bakery.

W. J. BOYD.

Effect on their bread-making properties of extracting flours with ether. A. H. JOHNSON (Cereal Chem., 1928, 5, 169—180).—Bread produced from ether-extracted flour was found to be better in colour, texture, and volume than that produced from natural flour. The improvement in loaf-volume was more marked with low-grade than with patent flours. The extensibility of ether-extracted flours was slightly lower than that of the corresponding natural flours, probably due to slowness of hydration of the particles of the former. The quality and quantity of the gluten, the absorption, and the viscosity of the water-extracted acidulated suspension were unaltered. Diastatic activity and reducing sugar content were higher in the ether-extracted flours. The differences in behaviour of the extracted and unextracted flours as regards colour and texture of the bread were not removed by adding lard to both. Treatment of the natural flour with 70% alcohol by doughing, air-drying, and grinding injured its baking properties more than treatment with 96% alcohol, and treatment with water was least injurious. The last-named only reduced the extensibility and viscosity without affecting the bread-making properties.

W. J. BOYD:

"Rope" in bread. E. A. FISHER and P. HALTON (Cereal Chem., 1928, 5, 192—208).—With decreasing p_H value of the bread, the development of rope is delayed. This value rises as the disease progresses owing to the production of ammonia. The buffering effect of the bread decreases till the disease is fully developed owing to the utilisation of the soluble phosphates, and thereafter it increases owing to the liberation of buffering material from proteins and other complex substances. Slight stickiness and faint odour are indicative of the acute stage of the disease. Of various substances tested, including acetic, lactic, and tartaric acids, and acid potassium and calcium phosphates, the most effective in preventing "rope" was the last-named, addition of 2 lb. of which per sack of flour was a complete preventative. Low temperature, ventilation, and cleanliness, however, are the best preventatives.

W. J. BOYD.

Obtaining crude lactose and other solids from sweet whey. R. W. BELL, P. N. PETER, and W. T. JOHNSON, JUN. (J. Dairy Sci., 1928, 11, 163—174).—Sweet whey (<0.2% acidity) is neutralised to p_H 7.3 with 5% sodium hydroxide solution, heated at 60°, evaporated to 62% of solids at 50°, cooled to 25°, seeded, and kept for 42 hrs. at 4° or 18 hrs. at 0°, the lactose being recovered with a centrifuge. The liquor yields on evaporation a soluble powder containing casein and lactalbumin (1 : 2).

CHEMICAL ABSTRACTS.

Nature of the microscopic grains in cheese. O. LAXA (Lait, 1927, 7, 521—525; Chem. Zentr., 1927, II, 1314).—The microscopic grains often found in cheese are shown to be composed of tyrosine or leucine. The presence of lactic acid in the early stages of the ripening of cheese increases considerably the solubility of tyrosine which then remains in solution, whilst leucine is precipitated in the anhydrous form during the final stages of the decomposition of the casein. In highly acid solutions leucine fails to precipitate, and in strongly alkaline media both tyrosine and leucine are soluble. A. R. POWELL.

Analytical evaluation of technical casein. F. BAUM (Chem.-Ztg., 1928, 52, 517—518).—The proportion of true casein substance obtained after deduction of the fatty acid, neutral fat, ash, and water content of a number of commercial caseins shows variations of 3—4%. The calculated nitrogen content of the true casein substance does not vary more than 0.6%, which is probably due to experimental error rather than to any difference in composition. So far no definite relationship has been revealed between the practical value of a casein and its composition.

F. R. ENNOS. ; }

Analyses of sprayed apples for lead and arsenic. A. HARTZELL and F. WILCOXON (J. Econ. Entomol., 1928, 21, 125—130).—The fruit of apple trees sprayed five times with lead arsenate (5 lb. per 150 gals.) contained (mg. per kg.): lead 0.55—1.80, average 0.912; arsenic as trioxide 0.042—0.193, average 0.099. The results are compared with those of the previous year.

CHEMICAL ABSTRACTS.

Rapid determination of vaseline oil in coffee. (Miss) E. A. GAUTHIER (Annali Chim. Appl., 1928, 18, 194—195).—The following modification of Marotta and Alessandrini's method (B., 1928, 314) is recommended. 25 g. of the coffee beans are extracted by prolonged agitation with about 150 c.c. of ether in two or three portions, the liquid being filtered and the ether distilled off on a water-bath. The brown residue, containing vaseline oil, fats, colouring matters, and resins, is saponified by heating for 15—30 min. in a reflux apparatus with 20 c.c. of 5% alcoholic potassium hydroxide. The alcohol is removed by evaporation and the dry soap dissolved in methyl alcohol, with gentle heating on a water-bath if necessary. The turbid methyl alcohol solution is transferred to a separating funnel, the flask being rinsed first with methyl alcohol (about 50 c.c.) and then with light petroleum, which is used to wash down the funnel and glass rod used. The separator is then vigorously shaken and, when the liquids have separated completely, the methyl alcohol solution is allowed to flow into the original flask, while the petroleum is washed with several small quantities of water and afterwards filtered into a tared dish or, if the solvent is to be recovered, a tared wide-necked flask. After distillation of the petroleum, the extraction of the methyl alcohol is repeated to remove any residual traces of vaseline. The total residue is dried for 30 min. at 100° and weighed. With coffee containing 0.5% of vaseline oil, the errors found ranged from 0.9 to 3.64%.

T. H. POPE.

Organic mercury compounds for the control of insects in stored seeds. J. L. HORSFALL (J. Econ. Entomol., 1928, 21, 147—150).

Determination of nitrogen. PARISI.—See XVI.

PATENTS.

Preservation of meat. H. WATKINS-PITCHFORD (B.P. 291,818, 10.1.27).—A method of injecting preservative saline solution into animal carcasses is claimed.

F. R. ENNOS.

Recovery of organic substances from [meat-pickling] liquors. LABORATORIUM "TASCH" A.-G. ["Tasch" LABORATORY, LTD.] (B.P. 286,208, 10.11.27. Ger., 28.2.27).—Liquors from the salting of meat are treated with organic sulphonic acids (e.g., guaiacol-

sulphonic acid) or sulphuric acid, which precipitate the albumins etc. as double compounds. B. FULLMAN.

Conditioning and drying grain. T. ROBINSON & SON, LTD., and C. J. ROBINSON (B.P. 291,881, 11.3.27).

[Continuous] method and apparatus for preparing cream fondant and the like. R. HEAD, C. CLAY, BAKER, PERKINS, LTD., and ANC. ETABL. A. SAVY, JEANJEAN & CIE. SOC. ANON. (B.P. 292,237, 15.3.27).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Pharmaceutical preparations of valerian. E. H. MADSEN (Dansk Tidsskr. Farm., 1928, 2, 177—183).—An examination of concentrated valerian preparations intended to give on dilution a product similar to the ordinary infusion of valerian. A suitable preparation can be made by percolation of the shredded root with 10% alcohol, any precipitate forming being collected on a filter after settling. Such an extract contains about 20.6% of the essential oil and is stable on keeping. Other extracts made according to the method of the Dutch Pharmacopœia (extraction of the macerated root once with dilute alcohol and then twice with water, the three extracts being united and evaporated to a syrup) and by a method in which the oil was first removed by distillation in steam and the distillate afterwards mixed with the extract of the residue, both yielded products containing about 50% of the essential oil originally present in the root employed.

H. F. HARWOOD.

Preservative action of "nipagin" [methyl *p*-hydroxybenzoate] and its homologues on pharmaceutical preparations. E. BÖHM and H. JEGLIŃSKI (Pharm. Ztg., 1928, 73, 480—481).—Materials which are easily susceptible to decomposition by micro-organisms, such as malt extracts, syrups, and some aqueous solutions, were treated with various preservatives, and kept for several months under conditions favourable for the development of bacteria. Nipagin and its methyl and ethyl homologues gave satisfactory results in all cases.

S. I. LEVY.

Simplified method for determination of silver [in medicinals]. G. KOGAN (Pharm. Zentr., 1928, 69, 228—229).—A weighed quantity of the material in a flask is treated on a water-bath with excess of nitric acid (*d* 1.2), nitrous acid is destroyed by means of permanganate, and the solution is diluted and titrated in the usual way with ammonium thiocyanate solution.

S. I. LEVY.

Examination of clove oil. H. W. VAN URK (Pharm. Weekblad, 1928, 65, 345—349).—The best test is the separation of the sodium eugenol compound by shaking with sodium hydroxide solution, but the quantity of the latter specified in the Dutch Pharmacopœia is too great; the best results are obtained with one tenth of the quantity. A modification of the phloroglucinol test is described.

S. I. LEVY.

PATENTS.

Manufacture of substituted glycol monoethers. CHEM. FABR. AUF ACTIEN (FORM. E. SCHERING), Assees. of W. SCHOELLER and H. SCHOTTE (G.P. 446,324, 26.3.24).—Unsaturated alcohols are treated with mercuric salts (acetate) in presence of an alcohol, and the products,

after conversion into halogen compounds, react with amines to give aminoglycol monoalkyl ethers. The process is particularly useful for manufacture of γ -amino- $\alpha\beta$ -glycol monoethers. Thus, allyl alcohol with mercuric acetate and methyl alcohol gives a mercuriacetate, which is converted successively into the mercuribromide, γ -iodo- β -methoxypropyl alcohol, b.p. 65—66°/0.4 mm., and γ -amino- β -methoxypropyl alcohol, b.p. 104—105°/10 mm. (phenylcarbamide described), or its *N*-methyl (b.p. 75°/12 mm.) and *N*-diethyl (b.p. 62—63°/1.2 mm.) derivatives; the piperidino-compound, b.p. 81—82°/0.3 mm., and quaternary trimethyl- γ -hydroxy- β -methoxyethylammonium iodide, m.p. 108—109°, are also prepared. From allyl alcohol, ethyl alcohol, and mercuric acetate the corresponding β -ethoxy-compounds are obtained: γ -iodo- β -ethoxypropyl alcohol, b.p. 78—80°/1.2 mm., gives with diethylamine γ -diethylamino- β -ethoxypropyl alcohol, b.p. 100—101°/12 mm., and with methylamine the γ -methylamino-compound, b.p. 89—91°/13 mm. (picrate, m.p. 92—93°). Cinnamyl alcohol yields a methoxymercuribromide, m.p. 110—111°, and an ethoxymercurichloride, m.p. 86°. From the former are obtained γ -iodo- β -methoxy- γ -phenylpropyl alcohol (not distillable even in high vacuum), which is converted by diethylamine into γ -diethylamino- β -methoxy- γ -phenylpropyl alcohol, which partly distils at 123—125° in high vacuum. *iso*Crotonyl alcohol behaves similarly. The products find therapeutic application.

C. HOLLINS.

Manufacture of a derivative of hexylresorcinol. F. HOFFMANN-LA ROCHE & Co. A.-G. (Swiss P. 117,236, 23.7.25).—4-Hexylresorcinol forms with hexamethylenetetramine a compound, m.p. 127—128°, having a pleasant taste and less irritating effect than hexylresorcinol, and being less sensitive to light and air.

C. HOLLINS.

Manufacture of *p*-carboxybenzenesulphon-dichloroamide. M. CLAASS (Swiss P. 118,115, 29.8.25).—*p*-Carboxybenzenesulphon-dichloroamide, m.p. 203°, obtained by treating *p*-carboxybenzenesulphonamide with 0.5*N*-sodium hypochlorite and adding dilute sulphuric acid until acid to Congo paper, is a powerful antiseptic and germicide.

C. HOLLINS.

Preparation of easily soluble derivatives of *m*-aminobenzoic esters. G. B. ELLIS. From CHEM. WORKS FORMERLY SANDOZ (B.P. 291,850, 4.3.27).—Readily soluble alkylsulphonates, which are unchanged by prolonged heating in aqueous solution at 100°, and are non-irritant local anaesthetics, are obtained by interaction of alkyl *m*-aminobenzoates with alkylsulphonic acids in absolute alcohol. Ethyl *m*-aminobenzoate gives a methanesulphonate, m.p. 149—150°, and an ethanesulphonate, m.p. 149—149.5°; *n*-propyl *m*-nitrobenzoate, b.p. 175—176°/17 mm., is reduced to the amino-ester, b.p. 176—177°/16 mm., which yields a methanesulphonate, m.p. 162—163°, and an ethanesulphonate, m.p. 150—151°; isobutyl *m*-nitrobenzoate, b.p. 180—181°/16 mm., is reduced to the amino-ester, b.p. 181—182°/17 mm., which gives a methanesulphonate, b.p. 178—179°, and an ethanesulphonate, m.p. 138—139°.

C. HOLLINS.

Manufacture of camphor solutions. I. G. FARBER-ENIND. A.-G., Assees. of M. BOCKMÜHL and R. SCHWABE (G.P. 446,290, 22.4.25).—Ethers or esters of polyhydric

alcohols are used as solvents for camphor intended for subcutaneous or intramuscular injection. Examples are glycol monoethyl ether, "diethylin," glycerol mono- and di-acetates, these being miscible in all proportions with water or serum.

C. HOLLINS.

Manufacture of ω -chloroacetylpyrocatechol [3:4-dihydroxyphenacyl chloride]. E. OTT (G.P. 445,524, 22.4.26).—Pyrocatechol, heated at 100° with chloroacetyl chloride (free from phosphorus compounds) in a solvent (benzene), gives a 40% yield of the *monochloroacetate*, m.p. 81°, which is converted by pure phosphoryl chloride, at 100° or in boiling benzene, into 3:4-dihydroxyphenacyl chloride, an intermediate for the synthesis of adrenaline (cf. Ott, A., 1926, 722). In the first stage of the reaction the *bischloroacetate* is obtained as by-product.

C. HOLLINS.

Manufacture of pyridine-2:3-dicarboxylic acid. GES. F. CHEM. IND. IN BASEL (Swiss P. 118,966, 11.8.25).—Quinolines containing in the Bz-nucleus at least one substituent (hydroxyl, amino-, halogen) favouring disruption by oxidation are oxidised with nitric acid or nitric and hydrochloric acids. Examples are: 8-hydroxyquinoline and its 5-chloro-derivative oxidised with 60% nitric acid at 70–90°, or by 60% nitric acid and concentrated hydrochloric acid at 70°, to give pyridine-2:3-dicarboxylic [quinolinic] acid.

C. HOLLINS.

Manufacture of derivatives of tertiary aromatic bases containing sulphur and phosphorus. L. CASSELLA & Co. G.M.B.H., Assees. of L. BENDA and W. SCHMIDT (G.P. 445,568, 3.1.25. Cf. B.P. 214,836; B., 1924, 615).—Tertiary aromatic amines are treated with sulphur and phosphorus trichloride, and afterwards with alkali, to give crystalline products useful as antirachitics and stimulants of metabolism. Dimethylaniline is heated with sulphur at 100° until the latter is dissolved, cooled to 50°, and warmed with phosphorus trichloride. The thick oily liquid is poured upon aqueous sodium hydroxide and ice, and aniline excess is removed by steam-distillation. Alcohol extracts from the evaporated residue "*p*-dimethylaminophenylthiophosphinic acid" (sodium salt + Aq, m.p. 97–98°). *N*-Ethylcarbazole, heated with sulphur and phosphorus trichloride at 120–130°, gives an analogous product (sodium and calcium salts described).

C. HOLLINS.

Manufacture of pure hexamethylenetetramine hydrochloride. PELLURIN-GES.M.B.H. (G.P. 445,646, 7.2.23).—Crude or purified hexamethylenetetramine, e.g., the base obtained by warming its crystallised hydrate at 30°, is dissolved in a non-aqueous solvent (high-strength alcohol, methyl alcohol, acetone, ethyl acetate, or chloroform) and dry hydrogen chloride is led in until no further precipitation occurs. The hydrochloride, $C_6H_{12}N_4 \cdot HCl$, is therapeutically more valuable than the free base.

C. HOLLINS.

Manufacture of compounds of 5:5-disubstituted barbituric acids with antipyrin. F. HOFFMANN-LA ROCHE & Co. A.-G. (Swiss P., 120,444–6, 9.5.25. Addns. to Swiss P. 116,465; B., 1927, 829).—The prior process is applied to the preparation of antipyretic and analgesic compounds from equimolecular proportions of antipyrin and (A) 5-phenyl-5-ethyl-, (B) 5:5-

diethyl-, (c) 5:5-diallyl-barbituric acids. The products have m.p. 112°, 118°, and 97°, respectively.

C. HOLLINS.

Manufacture of derivatives of 5:5-disubstituted barbituric acids. J. D. RIEDEL A.-G. (G.P. 445,670, 19.8.25).—Additive compounds, which crystallise readily from water and slowly dissociate at 15°, are obtained by adding a halogenated hydrocarbon to a 5:5-disubstituted barbituric acid, especially one containing an unsaturated substituent (allyl), melted under water. Additive compounds of chloroform with 5- β -bromoallyl- and 5-allyl-5-*sec*-butyl-barbituric acids, and of carbon tetrachloride with 5- β -bromoallyl-5-*sec*-butylbarbituric acid, are described; the last-mentioned has m.p. 125°. *iso*Propyl bromide may also be used.

C. HOLLINS.

Preparation of bismuth nucleinate. C. LEVADITI (F.P. 577,407, 19.2.24).—Sodium nucleinate dissolved in a solution of sodium chloride is heated for 6 hrs. at 37° with a solution of sodium potassium bismuthyl tartrate containing 30% Bi. Next day the crude bismuth nucleinate is precipitated by the addition of absolute alcohol. The precipitate is redissolved in sodium chloride solution and the liquid is subjected to dialysis through a collodion membrane suspended in a solution of sodium chloride. When no further bismuth passes through the membrane the colloidal solution is evaporated at a low temperature to a dry residue containing 32% Bi, 5% P, and 10% N. The compound may also be made directly from the solution obtained by extracting minced thyroid glands with sodium chloride solution.

A. R. POWELL.

Production of nitropyridinearsinic acids. A. BINZ and C. RÄTH (B.P. 275,590, 18.7.27. Ger., 5.8.26).—Hydroxypyridinearsinic acids, such as the 2:5-compound described in B.P. 250,287 (B., 1926, 512), are nitrated *ortho* to the hydroxyl group by mixed acid at 100°.

C. HOLLINS.

Tellurium compounds for germicidal and therapeutic purposes. G. T. MORGAN and H. BURGESS (B.P. 292,222, 14.3.27).—Claim is made for the preparation of the compounds described in A., 1928, 435. All the products find application as germicidal or therapeutic agents.

C. HOLLINS.

Preparation of double compounds of alkaloids or amines with antimony or arsenic tri-iodide. E. VIEL (F.P. 577,371, 14.4.23).—Acid solutions of alkaloids or of amines are mixed with acid solutions of arsenic or antimony tri-iodide containing an alkali or alkaline-earth iodide whereby amorphous precipitates are obtained which can be crystallised from hot dilute hydrochloric acid (cf. Caille and Viel, A., 1925, i, 68).

A. R. POWELL.

Manufacture of quinine solutions. F. B. DEHN. From CHEM.-PHARM. A.-G. BAD HOMBURG (B.P. 291,565, 4.4.27. Addn. to B.P. 266,209; B., 1927, 317).—The solubilising effect of terpenes on quinine base in olive and similar oils is enhanced by the addition of oil-soluble or fat-soluble alcohols, such as octan- β -ol, *n*-decyl alcohol, benzyl alcohol, β -phenylethyl alcohol, cinnamyl alcohol. Terpenes such as cineole, which have small solubilising effect but are therapeutically valuable, can be used. An example is dry quinine base (6 pts.),

benzyl alcohol (15 pts.), and cineole (15 pts.), made up to 100 pts. with olive oil. C. HOLLINS.

Preparation of benziminazolone-[5]-jarsinic acids. W. KOLLE, K. STREITWOLF, and A. FEHRLE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,674,368, 19.6.28. Appl., 19.7.26. Ger., 31.7.25).—See B.P. 256,243; B., 1927, 670.

Preparation of nitropyridinearsinic acids. A. BINZ and C. RÄTH (U.S.P. 1,675,402, 3.7.28. Appl., 28.7.27. Ger., 5.8.26).—See B.P. 275,590; preceding.

Manufacture of complex antimony compounds. H. HAHN, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,674,362, 19.6.28. Appl., 11.3.26. Ger., 12.5.25).—See B.P. 262,301; B., 1927, 125.

Production of carbohydrate-acridine compounds and solutions thereof. C. SCHNORF and F. HEFTI (U.S.P. 1,674,923, 26.6.28. Appl., 23.10.24. Ger., 12.4.24).—See B.P. 247,250; B., 1926, 341.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photochemistry of silver halides. I. Gravimetric determination of excess silver in photographic layers. H. H. SCHMIDT and F. PRETSCHNER (Z. wiss. Phot., 1928, 25, 293—307).—The gelatin of the thoroughly washed silver halide emulsion is destroyed by boiling with 1 : 30 nitric acid, a concentration which does not decompose the halides, prevents their reduction by gelatin and its decomposition products, and, in the case of iodide, causes no oxidation to iodate. The filtrate containing the excess silver and dissolved silver halides is treated with a known excess of potassium chloride, and the silver obtained by the reduction of the precipitated silver chloride with alkaline hydrazine sulphate is weighed (*a*). By treatment of the solution containing the excess chloride with a known excess of silver nitrate a weight (*b*) of silver is similarly obtained. The difference between the silver equivalent of the potassium chloride and the weight (*b*) gives the excess silver present in the solution, whilst the amount of dissolved silver halides is obtained by subtracting the excess silver from the value (*a*). The halides of the emulsion remaining after the treatment with nitric acid are shown to contain no excess silver or halogen by analysis, according to the above methods, of the liquid obtained from their purification by reprecipitation. Although all the emulsions studied were prepared in presence of excess halide, excess silver was found in each case, the amount being greatest for chloride and least for iodide, but bearing no relation to the method of preparation or the sensitivity of the emulsions. The work of Eggert, Noddack, and Leszynski, on the application of the photochemical equivalence law to silver halide layers, is criticised, since no account was taken of the excess silver present in the emulsions. J. W. GLASSETT.

Photochemistry of silver halides. II. Decomposition of photographic layers with neutral, acid, and alkaline hydrogen peroxide, and with ammoniacal ammonium sulphide. H. H. SCHMIDT and F. PRETSCHNER (Z. wiss. Phot., 1927, 25, 354—362; compare preceding abstract).—Acid hydrogen peroxide

increases the ease of decomposition by decomposing the gelatin, so that the concentration of the nitric acid can be reduced one hundredfold without decreasing the speed of decomposition. The solution can easily be filtered to a clear filtrate to give a residue of silver halide. The halogen ion in the filtrate can only be determined by means of silver nitrate if the hydrogen peroxide be first removed by long boiling of the ammoniacal solution. Neutral hydrogen peroxide completely decomposes the gelatin on boiling, and the silver halide can be collected on a filter. Some metallic silver is formed in the layer by reduction, but the equivalent quantity of halide in the filtrate can be determined as before. When an alkaline peroxide solution is used the gelatin is very rapidly decomposed, but the silver halide is reduced so that the residue contains metallic silver and the filtrate the halogen ion. The reduction is complete in the case of the chloride and bromide, but only partial with the iodide unless the process be repeated several times. Ammoniacal ammonium sulphide easily and completely transforms the silver into sulphide, and by addition of ammonia during washing of the precipitate, peptisation can be prevented and filtration thus facilitated. S. J. GREGG.

PATENTS.

Production of light-sensitive films. U. DIEM-BERNET (B.P. 271,507, 20.5.27. Switz., 21.5.26).—In a method for the preparation of transparent light-sensitive films from viscose, the silver halides are introduced during the manufacture of the film, by impregnating the film mass with soluble halide or silver salt, followed after superficial drying, if desired, by an adequate amount of silver salt or halide such that no opalescence is obtained upon complete drying. Such films are very suitable for reflex copying, and suffer little distortion during development and fixing. J. W. GLASSETT.

Manufacture of polychromatic screens for colour photography. R. C. M. DE BERCEGOL (U.S.P. 1,673,349, 12.6.28. Appl., 2.12.22. Fr., 5.12.21).—The film or plate is coated with a colouring agent, and, after excess has been removed, a layer of waxy insulating material is applied. Parallel lines are then cut through both waxy and colour layers, and the grooves so formed are filled with a second colouring agent. F. G. CLARKE.

Photographic sensitisers. KODAK, LTD. From EASTMAN KODAK Co. (B.P. 292,374, 28.7.27).—Alk-iodides or similar quaternary salts of 4-methyl- or 4 : 6-dimethyl-quinoline are condensed with iodoform (in place of formaldehyde) and alkali. In addition to the known sensitising dyes, there are formed under these conditions less soluble by-products which sensitise effectively from 780 to 900 μ , i.e., much further into the infra-red. C. HOLLINS.

Photographic developing processes more especially for use with printing surfaces. Soc. DE PHOTOCHIMIE ELKA (B.P. 265,630, 7.2.27. Fr., 8.2.26).—Gelatin reliefs suitable for all types of printing processes may be obtained from ordinary gelatino-bromide emulsions by partial development of the image in a hardening developer, such as pyrogallol, with a small amount of sulphite, followed by complete development, either in the darkroom or in daylight, with

an active non-hardening agent such as metol. Finally, after normal fixation, the negative is developed in hot water.

J. W. GLASSETT.

XXII.—EXPLOSIVES; MATCHES.

Pentaerythritol tetranitrate as a military explosive. A. STETTBACHER (Z. angew. Chem., 1928, 41, 716—718).—Pentaerythritol tetranitrate fulfils many of the requirements of a modern explosive, but because of its high m.p. (140—141°) must be used in a compressed state. When compressed it is much less sensitive, and is detonated with difficulty. This disadvantage is overcome by the use of a paste containing 80% of tetranitrate and 20% of nitroglycerol. Pentaerythritol tetranitrate may be prepared by adding 100 g. of the powdered alcohol to 430 c.c. of fuming nitric acid (d 1.52) and precipitating the ester by the careful addition of 400 c.c. of concentrated sulphuric acid. The temperature should not exceed 25°. The separated crystals are agitated with 1 litre of ice-water, filtered, and washed. Yield, 225 g. = 97%. The last traces of acid are removed by dissolving 100 g. of the crude ester in 400 c.c. of acetone and boiling under reflux with 3—4 g. of ammonium carbonate. The filtered solution is poured into 800 c.c. of water. The precipitated ester is collected, washed, and dried. Yield about 96% of the crude ester.

J. S. CARTER.

PATENTS.

Impregnation of absorptive material for use in explosives. Blasting explosive. B. I. STROOPS, Assr. to HERCULES POWDER CO. (U.S.P. 1,671,792—3, 29.5.28. Appl. [A] 2.4.27 and [B] 4.11.27).—(A) Ammonium nitrate or perchlorate is dissolved in the liquid obtained by heating a hydrated salt, such as magnesium or calcium nitrate or sodium sulphate, until it melts in its water of crystallisation, and a carbonaceous absorptive material, e.g., sawdust, is incorporated in the solution. (B) A blasting explosive comprises a mixture of 5—20% (10%) of nitroglycerin, 20—80% (47%) of ammonium nitrate, 4—30% (15%) of magnesium nitrate hexahydrate, 0—20% (12%) of sodium or potassium nitrate, and 5—30% (16%) of balsa wood sawdust.

A. R. POWELL.

Separation of 2:4-dinitrophenol and picric acid (F.P. 575,302).—See III.

XXIII.—SANITATION; WATER PURIFICATION.

Relative toxicities of arsenicals and fluorine compounds to various organisms. S. MARCOVITCH (J. Econ. Entomol., 1928, 21, 108—114).—Relative and quantitative toxicities of a number of arsenic and fluorine compounds for mosquitos, grasshoppers, worms, etc. are recorded. Toxic doses for man are: sodium fluoride 30 g., sodium fluosilicate 7.2 g., potassium arsenite 0.84 g.

CHEMICAL ABSTRACTS.

Electro-osmotic purification of water. A. H. W. ATEN (Chem. Weekblad, 1928, 25, 211—219; cf. Behrman; B., 1927, 958).—The apparatus manufactured by the Siemens Electro-Osmosis Co. of Berlin for the purification of water is described, and results are given of tests carried out. A current is caused to pass through a series of cells, each of which is divided into three parts

by means of two diaphragms. Electrodes are placed in the outer compartments. Electrolytes are removed from the water in the middle compartments as a result of differences in the resistances of the two diaphragms to the passage of cations and anions respectively. The theory of the process is discussed, and the results arrived at are interpreted to show that it is possible completely to free the water from dissolved electrolytes. Organic matter is also reduced, and the water rendered sterile.

S. I. LEVY.

PATENTS.

Activated sludge process of purifying sewage and other impure liquid. ACTIVATED SLUDGE, LTD., and J. A. COOMBS (B.P. 285,944, 27.10.26).—Sewage is subjected to preliminary agitation and aeration which breaks up grosser suspended solids, stops septic action, facilitates removal of grease, and, generally, renders sewage more amenable to subsequent treatment. After separation of solid matters and removal of grease in specially designed tanks the further treatment of the sewage is divided into two stages: (a) removal or oxidation of carbonaceous matter, (b) oxidation of ammoniacal matter to nitrite and nitrate, with the production and utilisation, in each case, of a special sludge for the work and consequent reduction in the total time involved.

C. JEPSON.

Manufacture of [disinfectants from] new complex bodies containing mercury. W. CARMAEL. From I. G. FARBENIND. A.-G. (B.P. 292,245, 16.3.27).—A soluble mercury salt is incorporated with a soluble arylaminesulphonate in presence of water without heating; mercuration is thus prevented (cf. B.P. 8153 of 1914; B., 1915, 852), but complex salts are formed from which mercury is not removed by sodium hydroxide or sulphide.

C. HOLLINS.

Removal of salts from sea water or salt water. R. J. WOOD (B.P. 285,954, 19.11.26).—Sea water or salt water is rendered safe for use in steam generators by treatment with appropriate reagents and the application of heat in a plant consisting of a reaction vessel, fitted with a steam jacket and agitators, and having valves so connected that they may be set and adjusted simultaneously so as to vary the proportion of salt water to reagent, followed by a decanter vessel in which is a filtering arrangement.

C. JEPSON.

Filtration of alkaline waters. J. GORDON. From R. E. HALL (B.P. 291,970, 28.6.27).—The use of various silicates (e.g., of iron, magnesium, or calcium) in preference to sand for filters dealing with hot boiler waters which are alkaline owing to the pretreatment they have received for softening purposes is recommended because of their lower solubility under such circumstances. For the filter bed a granulated smelter slag obtained from copper smelting may be used. As the silicates suggested have a fairly high sp. gr. the time taken to wash the filter by reversal of flow is not excessive.

C. JEPSON.

Refuse destructors. G. M. CLARK. From STETTNER CHAMOTTE-FABR. A.-G. VORM. DIDIER (B.P. 292,630, 22.1.27).

Base-exchange bodies (B.P. 279,466).—See VII. **Stabilisation of antiseptics** (G.P. 446,130).—See XVIII.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

AUG. 31, 1928.

I.—GENERAL; PLANT; MACHINERY.

Indicating equipment for industrial p_H measurements. H. C. PARKER (Ind. Eng. Chem., 1928, 20, 676—680).—When the concentration of acid or base exceeds 0.1% conductivity measurements are preferable to p_H measurement, provided the concentration of salts remains fairly constant. Tables are given for converting voltages into p_H values for hydrogen and quinhydrone electrodes at various temperatures. For many industrial purposes, however, the voltage is sufficient. The quinhydrone electrode becomes inaccurate above p_H 8.5, and above p_H 10 the hydrogen electrode should always be used. Three types of complete p_H indicators operating either or both of these cells are illustrated. Directions are given for making up the calomel cell and preparing the hydrogen and quinhydrone electrodes. The accuracy aimed at is 0.05 p_H unit. C. IRWIN.

Pfund colour grader. WILLAMAN. See XVII.

PATENTS.

Shaft furnace, gas producers, etc. STETTINER CHAMOTTE-FABR. A.-G. VORM. DIDIER, and E. TERRES (B.P. 292,052, 1.3.27).—The steam from the water-jacket of the furnace described in B.P. 284,639 (B., 1928, 352) is superheated by means of the gases from the same furnace, after they have passed through a dust-settling chamber. B. M. VENABLES.

Device for charging furnaces. WOODALL-DUCKHAM (1920), LTD., A. SCOTT, and S. N. WELLINGTON (B.P. 292,278, 23.3.27).—A hopper for charging loose and bulky material to a shaft furnace is caused to rotate, and is provided with a helical vane or flange on its interior surface. B. M. VENABLES.

[Open-hearth] furnace. A. G. EGLER (U.S.P. 1,674,167, 19.6.28. Appl., 25.10.26).—At each end of the hearth is situated a bridge wall which contains a gas port and an air chamber above the bridge wall; the air and gas uptakes are formed within a wall sloping downwardly and outwardly from the above-mentioned ports. B. M. VENABLES.

Heat exchanger. M. MAURAN, Assr. to MATHIESON ALKALI WORKS, INC. (U.S.P. 1,673,918, 19.6.28. Appl., 8.11.24).—Twin spiral passages are formed between double-spiral fins formed on a central body; the outer edges of the passages are closed by spiral ribbons. B. M. VENABLES.

Heat exchangers for use in catalytic apparatus. SYNTHETIC AMMONIA & NITRATES, LTD., and F. H. BRAMWELL (B.P. 292,404, 27.10.27. Addn. to B.P. 241,817; B., 1926, 12).—The heat exchanger of the previous patent, comprising double concentric tubes

wound into a spiral, has the single inner tube replaced by 3, 7, or other number of smaller tubes.

B. M. VENABLES.

Circulating apparatus for heating gases. CHEM. TECHN. GES.M.B.H. (B.P. 285,475, 19.1.28. Ger., 18.2.27).—In any apparatus using circulating hot gases, the burner for reheating the circulating gases is combined with the circulating fan. A preferred arrangement is a burner tube concentric in the outlet of the centrifugal fan, in the annular space round the burner tube are dampers to regulate the amount of returned old gases, and the excess old gas is discharged from the fan casing at a point or points behind the burner tube. B. M. VENABLES.

Drying apparatus for rotary kilns. F. LUTHER, Assr. to G. POLYSIUS (U.S.P. 1,675,717, 3.7.28. Appl., 26.5.27).—A horizontal, rotary cylinder has wide, internal, longitudinal baffles, the free edges of which are flexible, in order to scatter the material as it falls from these edges. The baffles are curved both longitudinally and transversely. F. G. CLARKE.

Rectifying apparatus. W. A. PETERS, JUN., Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,673,373—4, 12.6.28. Appl., 28.4.23).—(A) The liquid distillate from a condenser, attached to a rectifying column, flows through an adjustable double weir device by which a part of the liquid is run back to the rectifier and part elsewhere. (B) A rectifier is heated by steam at the lower part thermostatically controlled by the temperature higher up the column. A collecting tank for condensate is provided, and increase of the level of liquid in this decreases the supply of feed liquor and *vice versa*. B. M. VENABLES.

Machinery for grinding, mixing and like operations. J. H. J. WOOD (B.P. 291,952, 7.6. and 19.8.27).—A rotor revolves concentrically within a cylindrical housing. The rotor is formed with deep longitudinal flutes within which are imprisoned loosely grinding bars of various shapes of cross-section. The output and fineness are a function of the width and length of the annular grinding space. B. M. VENABLES.

Pulverisers. C. E. BRAINARD (B.P. 288,227, 15.11.27. U.S., 8.4.27).—A cylindrical mill is provided with grinding medium comprising a cylindrical roller subdivided into segments which may or may not fit exactly together. Feed or discharge is through hollow trunnions and the mill is lined with hard steel on all interior surfaces. B. M. VENABLES.

Centrifugal mills for the grinding, mixing, emulsification, and incorporation of solids and liquids, or liquids. C. P. WALKER (B.P. 291,957, 13.6.27).—Two discs, one or both rotating, are provided

with intercalating pins or blades, and the material is forced through the casing from the circumference inwards, *i.e.*, against centrifugal force. The blades may be sloped to promote, oppose, or be neutral to the passage of the liquid, and a further retarding means such as a valve may be placed on the outlet.

B. M. VENABLES.

Centrifugal machines. E. KNÜTTEL (B.P. 285,836, 21.2.28. Ger., 22.2.27).—A centrifugal separator, more particularly for finely-divided solids suspended in liquid, has its chamber or chambers formed in lenticular shape with an annular peripheral opening or slot of constant width. The slot is closed by a member which slides axially to the machine and is provided with grooves and projections which mate with the walls of the chamber in such a manner that accumulated solids form a seal. There are preferably two chambers on one driving shaft, the slide being arranged to close one and open the other.

B. M. VENABLES.

Separating or cleaning intermixed divided materials. K. DAVIS (B.P. 292,179, 13.12.26).—A perforated table through which air is blown with velocity decreasing in the direction of flow of the material is divided into zones of which the area is proportioned (a) to the proportion of impurities in any size range; (b) to the above proportion plus the proportion that size range bears to the whole quantity; or (c) to the above sum plus a further increase, the latter margin being greater in the finer sizes.

B. M. VENABLES.

Vacuum air separator. L. LINDSAY (U.S.P. 1,675,941, 3.7.28. Appl., 14.1.25).—The separator consists of a series of conical separating units connected in series, a grinding apparatus, and a blower, and has means for admitting gas to the upper zone of each separating unit, and for returning a portion of gas from the last unit to the grinding apparatus.

W. G. CAREY.

Air classifier. A. H. STEBBINS (U.S.P. 1,673,848, 19.6.28. Appl., 16.3.27).—An inclined cylindrical vessel is provided with longitudinal lifting blades and is rotated to shower the material within it. A current of air is produced up the incline by means of an external fan, and the air is simultaneously rotated by means of internal, separately driven, longitudinal fan blades having a large clearance between them and the blades on the wall of the rotating cylinder.

B. M. VENABLES.

Concentrator. A. H. STEBBINS (U.S.P. 1,673,849, 19.6.28. Appl., 6.5.27).—The flow of pulp is constrained to flow downwards through a narrow annular passage between a container and bulging core. The heavier particles continue along the side walls of the container which then converge to a bottom outlet. The lighter particles flow inwardly away from the walls and are discharged through a central outlet passing through the core.

B. M. VENABLES.

Strainers. A. E. WHITE. From ANDALE ENGINEERING Co. (B.P. 292,236, 15.3.27).—A twin strainer with change-over valves is described.

B. M. VENABLES.

Purification of colloidal liquids, emulsions, etc. M. G. W. HUMMELINCK (B.P. 291,810, 8.12.26).—The liquid is filtered through cloth or wire mesh, the aper-

tures of which are large compared with the colloidal or suspended particles, at a rate which does not exceed 1.8 m./hr., and under such conditions that the deposit is not disturbed.

B. M. VENABLES.

Separation of gases or vapours from gaseous mixtures. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 292,204, 10.12.26).—Two or more adsorbents of different structures, one at least having hydrophilic properties, and the first preferably having wide pores and successive adsorbents having fine pores, are used in the adsorption, *e.g.*, of benzene from coal gas or of hydrocarbons from hydrogen chloride. Portions of the gaseous mixtures may also be separated by other means before or during adsorption, *e.g.*, by compression, cooling, etc. [Stat. ref.]

L. A. COLES.

Extinguishing fires. EXCELSIOR FEUERLÖSCHGERÄTE A.-G., and O. TREICHEL (B.P. 280,543, 7.11.27. Ger., 15.11.26).—Liquid carbon dioxide and liquid carbon tetrachloride are mixed and the crystallised double compound formed is applied to the fire.

W. G. CAREY.

Refrigerating apparatus of the absorption type. H. D. FITZPATRICK. From S. K. D. M. VAN LIER (B.P. 293,525—6, 18.5.27).

Refrigerant evaporators. BRIT. THOMSON-HOUSTON Co., LTD. From GEN. ELECTRIC Co. (B.P. 293,556, 22.6.27).

Heat-interchange device. C. R. HOUSEMAN, Assr. to AIR REDUCTION Co. (U.S.P. 1,677,777, 17.7.28. Appl., 23.1.26. U.K., 28.1.25).—See B.P. 251,331; B., 1926, 516.

Producing in a solution the crystallisation of the body dissolved therein. A. DELAS, Assr. to SOC. DES CONDENSEURS DELAS (U.S.P. 1,677,551, 17.7.28. Appl., 25.7.25. Fr., 6.8.24).—See G.P. 412,968; B., 1925, 699.

Chemical apparatus (B.P. 293,077).—See X.

II.—FUEL; GAS; TAR; MINERAL OILS.

Ignition of solid fuels. H. H. GREGER (Brennstoff-Chem., 1928, 9, 232—234; cf. B., 1927, 864).—Apparatus for the determination of the ignition point of coke etc. consists of a glass tube (diam. 20 mm.) containing the sample (10—20-mesh), and immersed in an iron crucible full of sand in such a way that a current of air (1 litre/min.) can be drawn down through the heated sand in the crucible and up through a short column of sand within the tube and finally through the fuel sample. In this way the temperature of the latter can be raised rapidly and regularly by internal heating. The ignition point is shown by a sudden acceleration in the rise of temperature registered by a thermometer, the bulb of which is immersed in the fuel sample. The ignition points determined vary from 296° for a wood charcoal to 690° for a metallurgical coke. The influence of some physical factors on the phenomena accompanying ignition is discussed.

A. B. MANNING.

Reactivity of fuels. C. QUILLARD (Compt. rend., 1928, 187, 122—124).—It is suggested that the velocity of luminous combustion in a vertical column of solid fuel may be taken as a measure of its reactivity. The reactivities of various fuels have been determined by burning in a current of oxygen in a vertical quartz tube.

C. W. GIBBY.

Formation of gaseous and liquid hydrocarbons by the action of steam and of alkali on semi-coke under increased pressure. F. FISCHER and H. PICHLER (*Brennstoff-Chem.*, 1928, 9, 200—206).—When semi-coke, previously freed from tar by heating for 15 hrs. at 520°, is heated at 500° with water, preferably in the ratio 8:1, gas is evolved in quantity depending on the source of the semi-coke; after 3 hrs. the yield may vary from 350 c.c. to 2830 c.c. The composition is, however, practically constant. Besides 33—43% CO₂ there are present about 20% H₂ and 40% CH₄, the ratio of these constituents corresponding closely with that calculated by Reinders (cf. B., 1928, 5) for "ideal" water-gas produced at 380° and atmospheric pressure. The use of more water than 12.5% leads to a decreased yield of gas in which the hydrogen and methane contents are respectively increased and diminished. The use of less water also gives a lower yield of gas. When the reaction is continued longer than 3 hrs. the yield of gas is increased, but not proportionally. This is due to a diminution in the reactivity of the semi-coke. The hydrogen and methane contents are, however, decreased and increased, respectively, by longer heating. The reactivity of the semi-coke remains low when the reaction mixture is reheated after removing the gas produced. No appreciable improvement is effected by adding more water or by drying the residual semi-coke in air or at 350° *in vacuo* before reheating. Partly used semi-coke rapidly gives a large yield of gas, free from carbon dioxide, when heated with 5*N*-potassium hydroxide solution. This can be repeated, whereby much gas, richer in methane and poorer in hydrogen, is obtained if more alkali is added, or less gas, poorer in methane and richer in hydrogen, if the residual mixture is merely reheated after removing the gas previously formed. In general, the gas so obtained is rich in hydrogen rather than in methane. The addition of a large excess of solid potassium hydroxide gives a large yield of gas, free from carbon dioxide, and containing up to 44% of methane and 10% of higher gaseous hydrocarbons. A small yield (3—3.5%) of liquid hydrocarbons free from phenols is also obtained. Rubidium, potassium, and sodium hydroxides in 5*N*-solution produce effects of the same order of magnitude. Ammonia and milk of lime have but slight effect in promoting the reaction. Ammonium carbonate, ammonium sulphide, and sodium sulphide have no appreciable action; sodium and potassium carbonate in 10*N*-solution produce an appreciable yield of gas, but are less effective than the hydroxides. Iron, nickel, ferric oxide, nickel sulphate, cobalt chloride, and nickel and cobalt hydroxides are without influence on the reaction between semi-coke and water.

W. S. NORRIS.

Hydrogenation of coal in presence of catalysts. B. HLAVICA (*Brennstoff-Chem.*, 1928, 9, 229—231).—The effect of the addition of some metallic oxides and chlorides on the hydrogenation ("berginisation") of coal has been studied, the experiments being carried out in a rotating autoclave of 1.8 litres capacity. The most effective catalysts were the oxides and chlorides of zinc, nickel, cobalt, and copper; compared with ferric oxide, they increased the tar yield by 30—100%, shortened the reaction time to about one third, and improved the

quality of the oil. They did not, however, permit any lowering of the optimum temperature or pressure. Their action affects primarily the splitting up of the coal "molecule" and only secondarily the actual hydrogenation process.

A. B. MANNING.

Arsa [Istria] coals. M. G. LEVI and C. PADOVANI (*Annali Chim. Appl.*, 1928, 18, 245—272).—A note on the distillation and berginisation products.

Determination of water in combustible materials by means of magnesium methyl iodide. A. TAUBMANN (*Z. anal. Chem.*, 1928, 74, 161—167).—The magnesium methyl iodide method of determining the moisture in combustible materials by measuring the volume of methane evolved on treating the pyridine extract of the sample with the Grignard reagent is shown to give excellent results with coal, coke, bituminous shales, peat, and petroleum products. Several determinations can be carried out in 1 hr. and no heating is required.

A. R. POWELL.

Low-temperature carbonisation of lignites and sub-bituminous coals. J. D. DAVIS and A. E. GALLOWAY (*Ind. Eng. Chem.*, 1928, 20, 612—617).—Comparative assay tests at low temperature were made on twenty-four American coals and lignites by the oil-shale method (cf. Gavin, B., 1923, 299 A), and those of Gray and King (B., 1921, 205 A), and of Franz Fischer (*Z. angew. Chem.*, 1920, 33, 172). The oxygen content of the coals was above 19% and no coherence of the coke was observed. Only 6—20 gals. of tar per ton were obtained, and though the gas was normal in quantity the quality was poor. Agreement between the assay results obtained by the three methods was not close, and whilst it is believed that all three methods may give reasonably accurate indications in the hands of experienced workers, a preference is shown for the Fischer method.

C. A. KING.

Problems of the brown coal low-temperature carbonisation process. P. ROSIN (*Brennstoff-Chem.*, 1928, 9, 182—184).—The problems awaiting solution, such as the increase in the output of the ovens, the carbonisation of finely-divided brown coal, carbonisation by retort or low-temperature carbonisation gas, disposal and utilisation of the coke from brown coal, purification of the effluent liquors, the working-up of the tar with special reference to its dust content, and the use of the gas for long-distance supply systems, are discussed.

A. E. MITCHELL.

Wood as fuel. L. A. WANDERLEY (*Bol. Soc. Chim. São Paulo*, 1928, 1, 73—77).—Determinations were made of the calorific value and moisture content of the wood of *Eucalyptus* species and of native Brazilian trees. An attempt to imitate practical conditions by removing 25% of the moisture before determination of the calorific value gave discordant results. Determinations made on completely dried horizontal sections showed that all the woods examined had sensibly equal calorific value—about 4700 kg.-cal./kg., independent of source or age.

R. K. CALLOW.

Applications of chemistry in gas-making. H. HOLLINGS (*Gas J.*, 1928, 182, 924—935).—Examples are given in connexion with the free-carbon content of

tars from various sources, with the light spirit recovered by stripping coal gas, and with the use of a waste-heat boiler as a meter for waste gases. Recent calculations of leakage from retorts, by use of waste-gas analyses, indicate how serious such losses may be, and further use of gas analyses is made in relation to study of the simultaneous production of coal gas and water-gas in vertical retorts. A relation has been found between the coal gas therm yield and the calorific value of the inert-free coal gas which is expressed in the form of a nomogram or a slide rule, and enables close study to be made of working results. Ammonia recovery is discussed in the light of new data on equilibria between foul gas and liquors of varying strength, and a system of fractional condensation based on these results is described, which reduces the volume of liquor to a minimum. In the manufacture of water-gas an approximate method for independent calculation of coke and oil figures is illustrated, and results obtained on the efficiency of oil cracking under different conditions are briefly mentioned.

R. H. GRIFFITH.

Possibilities of increasing the gas efficiencies in water-gas and producer-gas manufacture. J. GWOSDZ (*Brennstoff-Chem.*, 1928, 9, 184—188).—The effect on gas efficiencies, from water-gas and producer-gas generators, of changing the working temperatures and pressures is discussed, with relation to the literature. The possibility of increasing the reactivity of the coke by use of a suitable catalyst is also considered.

A. E. MITCHELL.

Dust and moisture control [in gas mains]. K. C. TOMLINSON (*Gas Age Rec.*, 1927, 60, 811—814, 817—818).—Much of the dust in gas distribution lines is due to corrosion. The most corrosive constituent of the gas is hydrogen cyanide. Dehydration of the gas, or coating the pipe with oil, minimises the corrosion.

CHEMICAL ABSTRACTS.

Glass receiver for determination of benzene and benzines by means of active charcoal. A. WEINDEL (*Brennstoff-Chem.*, 1928, 9, 234).—A U-shaped calibrated receiver of improved design is described for collecting the benzene distilled from active charcoal in a current of superheated steam. It has been designed for use in conjunction with an apparatus for determining benzene in gas (B., 1927, 435).

A. B. MANNING.

Action of sulphuric acid on aromatic hydrocarbons in connexion with their detection in petroleum. M. D. TILICHEV and A. I. DUMSKI (*Neft. Choz.*, 1927, 13, 647—658).—Benzene, *p*-disubstituted benzene, and tetrasubstituted benzene are very resistant to sulphuric acid, but all other aromatic compounds present in petroleum are easily sulphonated; 95.6% and 91—92% acid, respectively, are required. Fuming acid reacts with naphthenes.

CHEMICAL ABSTRACTS.

Properties of sulphonic acids from petroleum products. A. DOBRJANSKI and B. ANUROV (*Neft. Choz.*, 1927, 13, 200—204).—Petroleum sulphonic acids are colloids. Salts of first-group metals, except copper, are soluble in water; beryllium, zinc, cadmium, and mercury salts are appreciably soluble in hot water. Arsenic, antimony, and bismuth do not form precipi-

tates. The acids are easily displaced from their aqueous solutions by salts and mineral acids.

CHEMICAL ABSTRACTS.

Composition of petroleum and its products. G. A. BURRELL (*Ind. Eng. Chem.*, 1928, 20, 602—608).—A review of the present state of knowledge of the more important phases of petroleum chemistry.

H. S. GARLICK.

Kerosenes from Grozni crude oil. A. I. VORONOV (*Neft. Choz.*, 1928, 14, 52—67).—Aromatic hydrocarbon contents of kerosenes are: Grozni 16, Baku 15, Emba 5, Maikop 29, California 7%. The 200—300° fraction is smaller in Russian than in Californian kerosene.

CHEMICAL ABSTRACTS.

Comparison of Soviet and foreign lubricating oils. II. S. S. NAMETKIN and B. T. ARKHANGELSKI (*Neft. Choz.*, 1927, 13, 642—646).—Foreign oils are darker, have a higher carbon content, and have f.p. from —5° to —19° compared with —8° to —18° for Soviet oils. The latter are probably less readily oxidised at high temperatures.

CHEMICAL ABSTRACTS.

Viscosimetry of lubricating oils. I. Wo. OSTWALD and A. FÖHRE (*Kolloid-Z.*, 1928, 45, 166—179).—Measurements of the viscosity of lubricating oils have been made at various pressures between 7.5 and 103.0 cm. of water. Seventeen different mineral oils, having viscosities both greater and less than that of glycerol, were examined at 25°, and with the exception of two Voltol oils, the Hagen-Poiseuille law was valid for each case. There is very little, if any, structure viscosity in the liquids examined, and the supposed similarity between lubricating oils and colloid systems is not borne out by these experiments. The Voltol oils have a smaller temperature coefficient than the other oils examined. In an examination of the influence of temperature on viscosity, good results were obtained by application of Schwedhelm's formula, which may be generalised to the form $z/G = (z'/G)^{t(t' - t)}$, where z and z' are the viscosities at temperatures t and t' , G is a constant for all oils and for the particular viscosimeter employed, and H is a characteristic constant for each oil.

E. S. HEDGES.

Degree of wetting of silica by crude petroleum oils. F. E. BARTELL and F. L. MILLER (*Ind. Eng. Chem.*, 1928, 20, 738—742).—The "adhesion tensions" of different crude oils for silica are calculated from the results of experiments on the displacement of the oils from silica by water (cf. B., 1928, 1). The factors governing the removal of oil from oil-bearing sand by means of "water-flooding" methods are discussed.

S. S. WOOLF.

Tabulated analyses of [319] representative crude petroleum of the United States. N. A. C. SMITH and E. C. LANE (*U.S. Bureau of Mines Bull.* 291, 1928; 69 pp.).

Organic compounds and ammonia from water-gas. BRUTZKUS.—See III. **Solubility of lubricating oil in liquid carbon dioxide.** QUINN.—See VII. **Carbon blacks and rubber.** GOODWIN and PARK.—See XIV.

PATENTS.

Manufacture of briquettes. P. JUNG (U.S.P. 1,675,272, 26.6.28. Appl., 28.7.22. Ger., 24.5.21).—A uniform mixture of a solid fuel with a small amount of a solid fuel material ground to a colloidal state of fineness is compressed in the form of briquettes.

A. B. MANNING.

Manufacture of liquid fuel. H. G. C. FAIRWEATHER. From AMER. COAL-OIL CORP. (B.P. 292,673, 22.3.27).—A solid fuel is finely powdered and the impurities are removed by froth flotation or agglomeration. The oil already admixed with the fuel in the cleaning process, or, if necessary, additional oil added subsequently, is thickened by oxidation with air in order to produce a stable suspension of the fuel.

A. B. MANNING.

Coking retort oven. J. BECKER, Assr. to KOPPERS Co. (U.S.P. 1,675,687, 3.7.28. Appl., 26.11.20. Renewed 18.6.26).—The regenerators of a coke-oven battery are arranged below and parallel with the coking chambers, and are grouped into pairs crosswise of the battery. They are connected with the vertical combustion flues of the heating walls. Reversing valve connexions are provided between a producer-gas main and alternate pairs of regenerators, and between the intermediate pairs of regenerators and the outer air. A pair of waste-gas tunnels is located on the same side as the producer-gas main, with valve connexions between one of the tunnels and the regenerators on the pusher side and between the other tunnel and the regenerators on the coke side of the oven. Controlling valves are provided for connecting one of the tunnels to the stack while the other is shut off therefrom.

A. B. MANNING.

Wet process for extinction of coke. SOC. GÉN. DE FOURN. À COKE SYSTÈMES LECOQ (B.P. 284,721, 3.2.28. Belg., 5.2.27).—The hot coke is sprayed with water in a closed chamber, and the steam produced is passed through a second chamber wherein a water shower condenses part of it and removes the suspended coke dust.

A. B. MANNING.

Manufacture of high-purity carbon. D. GARDNER (B.P. 292,798, 2.8.27).—Carbonised material, *e.g.*, wood charcoal, is ground to an impalpable powder, and is then washed successively with a hot alkaline solution (6*N*-sodium hydroxide) and hot nitric acid (*d* about 1.33). The washed material is finally heated at 1000–1300°. The alkaline treatment may be omitted and the acid treatment then followed by fusion with sodium or potassium bisulphate and subsequently washing with water. Organic solvents also may be introduced into one or other of the washing treatments. To protect the product against absorption of water it may be incorporated in turpentine, tetrachloroethane, etc.

A. B. MANNING.

Manufacture of activated carbons and decolorising charcoals. J. VAN LOON (B.P. 292,213, 10.3.27).—Activated carbon is prepared by grinding charcoal, or other material containing carbon, at a raised temperature, and, if necessary, under pressure, with a wetting agent, *e.g.*, water, alcohol, benzene, etc., which has also

a solvent action on the substances other than carbon in the starting material.

A. B. MANNING.

Gas producer. E. GOUTAL and H. HENNEBUTTE (B.P. 266,353, 16.2.27. Fr., 16.2.26).—A producer designed for supplying internal-combustion engines has a hearth of relatively small dimensions. The oxidation zone is provided with circularly disposed inlets for the injection of air, and is maintained as small as possible by the use of fuel of uniform character. The gas is enriched by the introduction of heavy oils into the reducing zone, which may be provided with a stack of refractory material to facilitate cracking. Steam may be injected into either or both zones.

A. B. MANNING.

Manufacture of coal gas. T. F. CANNING and R. G. CLARK (B.P. 292,235, 15.3.27).—A plant for the carbonisation of coal comprises a number of horizontal or inclined retorts and a vertical chamber, the upper part of which is directly connected with the discharge ends of the retorts. The retorts and the lower part of the chamber are heated by gas from a separate producer. The main carbonisation takes place in the retorts, the hot coke being then discharged into the vertical chamber, up which superheated steam is passed. The water-gas so produced passes through the retorts, assisting in the carbonisation of the charge and diminishing the cracking of the volatile products by sweeping them rapidly from the retort. The residual coke, cooled by the steam, is finally discharged from the lower end of the vertical chamber. If desired, oil for enriching the gas may be injected into the chamber.

A. B. MANNING.

Low-temperature distillation furnace. J. PLASS-MANN (B.P. 275,546, 6.1.27. Ger., 5.8.26).—A large number of minor modifications are introduced into the design of the apparatus described in B.P. 240,800 (B., 1926, 228), including a distributing device for feeding the heating gas uniformly to the separate cells, insulated vertical return shafts for the outflowing heating gases, a staggered arrangement of the filling and discharging devices in the distillation chambers, with corresponding filling shafts fed from a distributing bunker, improved coke discharging and quenching arrangements, etc.

A. B. MANNING.

Gasification of fuel. G. SZIKLA and A. ROZINEK (B.P. 274,110, 8.7.27. Ger., 9.7.26. Addn. to B.P. 255,857; B., 1927, 180).—The bottom of the gasifying chamber of the prior patent forms with the horizontal an angle smaller than the angle of rest of the incandescent fuel dust, so that the latter collects in the form of a crater surrounding the opening through which the air for combustion passes. Adjustable poking devices feed the settled dust towards the opening, which is provided with a slide valve; both valve and poking devices may be automatically controlled by the load on the plant, *e.g.*, by the boiler pressure if the plant is used for the production of steam power.

A. B. MANNING.

Distillation of solid carbonaceous material. M. J. TRUMBLE (U.S.P. 1,674,420, 19.6.28. Appl., 11.9.23).—The material is preheated in a current of steam, then subjected to destructive distillation by passing superheated steam through it under pressure, and finally cooled in a current of saturated steam, the

latter being then raised in temperature and utilised in the first stage of the process. A. B. MANNING.

Separation of coke or half-coke from dry distillation gases. TROCKNUNGS-, VERSCHWELUNGS-, U. VERGASUNGS-GES.M.B.H. (B.P. 285,387, 14.2.28. Ger., 14.2.27).—The separation of suspended coke dust from distillation gases, particularly in processes in which powdered coal is carbonised in a current of gas, is brought about by passing the dust-laden gas through a slot of which one wall remains stationary while the other moves. A convenient form of apparatus consists of a flat, cylindrical chamber in which a disc rotates. The suspended particles are driven towards the stationary wall, where they collect and whence they can be discharged by any suitable means. A. B. MANNING.

Absorbing hydrogen sulphide or hydrogen sulphide and ammonia from gases. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 292,669, 21.3.27).—Coal gas, coke-oven gas, etc., after complete or partial removal of the ammonia, are scrubbed successively with thiosulphate solution containing sulphurous acid and then with the polythionate solution obtained thereby mixed with sufficient of the previously recovered ammonia to maintain a neutral or weakly alkaline reaction in the liquor. Methods of working up the products and special applications of the process are described.

L. A. COLES.

Desulphurisation of gases. E. RAFFLOER, ASSR. to W. E. LEUCHTENBERG (U.S.P. 1,672,778, 5.6.28. Appl., 11.6.26.).—A portion of the gas to be desulphurised is used to blow the powdered purifying agent into a chamber where it meets and mixes with the main current of gas. The mixture passes on to a separator, where the purified gas is freed from solid, *e.g.*, by means of baffles and louvres, the solid being returned to the injector or renewed.

C. HOLLINS.

Production of carbon dioxide. F. H. KELLIHER (U.S.P. 1,675,497, 3.7.28. Appl., 19.6.26).—Finely-divided carbonaceous material is burned, in the presence of sufficient air, to carbon dioxide in a closed furnace. A piston pump sucks the gas through a scrubber and settling chamber, and passes it to a separator and gas-receiver. The water removed from the gas in the separator is introduced into the top of the scrubber.

F. G. CLARKE.

Burning of fuel. E. W. CLARKE, ASSR. to AMER. TAR PRODUCTS Co. (U.S.P. 1,660,831, 28.2.28. Appl., 13.10.22).—Pulverised pitch is forced, by means of a blast of air, through a delivery nozzle situated outside the furnace, with such velocity that it is maintained in a compact stream at the point of entrance into the furnace, and finally reaches the combustion zone before becoming adherent.

F. R. ENNOS.

Combustion apparatus. O. BRUNLER (B.P. 292,736, 25.4.27).—In order to prevent the flame travelling backwards within a submerged-flame burner an interchangeable reducing piece is inserted between the fuel valve and a bridge piece placed near the mouth of the burner.

A. B. MANNING.

Manufacture of liquid hydrocarbons and their derivatives, particularly those of low b.p. from

coal, tars, mineral oils, etc. I. G. FARBENIND. A.-G. (B.P. 272,556, 13.6.27. Ger., 14.6.26).—The initial materials are treated with hydrogen at a high temperature and under pressure, with or without a catalyst, in a current of the gas, preferably in closed circulation, in such large excess that the partial pressure of the products of low b.p. does not exceed 10% of the total pressure.

A. B. MANNING.

Distillation of emulsified [mineral] oils. G. EGLOFF and H. P. BENNER, ASSRS. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,674,819, 26.6.28. Appl., 20.8.20. Renewed 24.4.28).—Oil containing emulsified water is distilled by applying heat to the upper surface of the oil in the still, progressively lowering the point of application of heat, and finally heating the still externally by means of a furnace.

C. O. HARVEY.

Continuous distillation and condensation of hydrocarbons. A. CLARKE, ASSR. to UNITED HYDROCARBONS Co. (U.S.P. 1,674,852, 26.6.28. Appl., 9.8.21).—A mixture of gases and vapours, recovered by absorption, is removed from the absorbent medium by distillation and subsequently separated into a gaseous and a liquid fraction by compression and cooling. The vapours and gases are reheated and again passed to the compressing and cooling zones after combining with a further quantity of the vapours to be fractionated.

C. O. HARVEY.

Mineral oil still. A. E. PEW, JUN., ASSR. to SUN OIL Co. (U.S.P. 1,674,918, 26.6.28. Appl., 27.2.26).—A horizontal cylindrical still is reinforced internally by circumferential beams connected by transverse members with a central longitudinal reinforcement.

C. O. HARVEY.

Refining low-boiling distillates of earth and mineral oils. S. STRANSKY and F. HANSGIRG (B.P. 267,959, 17.3.27. Austria, 18.3.26).—Hydrocarbon fractions of low b.p. are freed from highly unsaturated compounds, which may cause resinification and bad odour, by treatment with about 5% of anhydrous aluminium chloride at room temperature, the mixture being cooled if necessary to maintain the temperature below 35°. Very little loss due to polymerisation of non-objectionable hydrocarbons occurs. After separation of the aluminium chloride the product is fractionally distilled.

C. O. HARVEY.

Refining of liquid hydrocarbons. H. WADE. From SILICA GEL CORP. (B.P. 292,231, 14.3.27).—Gasoline, kerosene, benzol, etc. are refined by means of silica gel at 121–149° to meet various specifications as follows: Removal of elementary sulphur is accomplished by the simple gel treatment; other sulphur compounds are removed by means of gel impregnated with up to about 1% of metallic oxides, such as those of copper and/or iron; and for removal of gum-forming constituents the gel is impregnated with about 5% by wt. of sulphuric acid.

C. O. HARVEY.

Fractionation of hydrocarbons. W. K. LEWIS and A. A. WELLS, ASSRS. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,672,849, 5.6.28. Appl., 5.3.24).—Kerosene hydrocarbons are separated from a gas-oil distillate by fractional distillation at about 30 mm. pressure, the vapours being passed through progressively cooler zones

where they meet a countercurrent of the condensate produced in the coolest zones. C. HOLLINS.

Cracking of [petroleum] oil. W. R. HOWARD, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,674,827, 26.6.28. Appl., 17.9.20. Renewed 24.4.28).—The oil to be treated is forced into the still by means of a steam pump situated inside the still. The exhaust steam from the pump discharges into the still. C. O. HARVEY.

Distillation of oils with catalysts. G. L. PRITCHARD and H. HENDERSON, Assrs. to GULF REFINING Co. (U.S.P. 1,672,339, 5.6.28. Appl., 28.11.21).—In the treatment of petroleum oils with aluminium chloride to obtain lower-boiling oils, the catalyst is more easily recovered in the earlier stages and long before its activity is exhausted. Accordingly the treatment is stopped after 0.1–5 gals. of distillate have been obtained per lb. of aluminium chloride, and the latter (liquefied by the oil) is run off. A highly active aluminium chloride–oil mixture can be recovered by distillation at 200–300°.

C. HOLLINS.

Apparatus for treating [cracking] hydrocarbon oils. F. E. WELLMAN (U.S.P. 1,672,668, 5.6.28. Appl., 20.3.23).—The oil is heated in a number of L-shaped tubes in a vertical, baffled furnace, the short limbs projecting laterally through the furnace walls, and the upper part of the long limbs being connected with several vapour drums outside the furnace. From the drums cracked vapour is drawn off to condensers, whilst liquid oil is recirculated to the short limbs of the L-tubes, from which residual oil is removed from time to time, and into which fresh oil is fed.

C. HOLLINS.

Treatment of hydrocarbons. L. DE FLOREZ, Assr. to TEXAS Co. (U.S.P. 1,674,390, 19.6.28. Appl., 4.8.22).—Oil is vaporised and the vapours are passed through a cracking zone, the velocity of flow being progressively increased. The vapours remaining after reduction of the velocity of flow by expansion are condensed.

C. O. HARVEY.

Clarification of benzine. J. J. WACK (B.P. 292,433, 13.1.28).—In the regeneration of benzine used for dry-cleaning processes, the removal of fatty matter is effected by centrifuging followed by saponification with a solution, into which it is sprayed, of sodium carbonate containing sodium chloride to assist demulsification and an alkaline hypochlorite. The alkaline solution is covered with a layer of viscous material such as glue, which acts as a filter and prevents any boiling of the benzine on the surface.

C. O. HARVEY.

Regeneration or purification of lubricating oils. SWAN, HUNTER, & WIGHAM RICHARDSON, LTD., and H. J. YOUNG (B.P. 292,300, 12.4.27).—Oils drawn from the lubricating systems of internal-combustion engines, while yielding no indications of the presence of corrosive substances when examined by the usual chemical tests, were found to contain sulphuric acid and metallic sulphates detectable by digestion with caustic soda or potash. These substances are removed (or replaced by harmless substances) by contact with solids in suitable form consisting of metallic zinc, aluminium, or magnesium, and their alloys with one another or with calcium and/or sodium.

C. O. HARVEY.

Production of oxidation products from gaseous hydrocarbons. J. H. JAMES, Assr. to C. P. BYRNES (U.S.P. 1,675,029, 26.6.28. Appl., 21.11.16. Renewed 28.3.25).—Products of incomplete combustion are obtained by passing hydrocarbon–oxygen mixtures (in which the proportion of the former is above the explosive limit) through a catalytic layer containing a compound of an electronegative metal of high m.p. and low atomic volume.

C. O. HARVEY.

Purification of montan wax. J. Y. JOHNSON. From I. G. FARBENTIND. A.-G. (B.P. 292,298, 8.4.27).—Crude or pretreated montan wax is separated from resin and fatty acids etc., and a product consisting of practically pure wax esters is obtained by extraction with hot glacial acetic acid.

C. O. HARVEY.

Bituminous emulsions. ASPHALT COLD MIX, LTD., F. LEVY, and L. G. GABRIEL (B.P. 292,251, 17.3.27).—The procedure of B.P. 202,021 (B., 1923, 1013 A) is modified by the substitution of a solution of an alkali silicate or borate for that of the hydroxide or carbonate.

A. B. MANNING.

[Apparatus for] burning of pulverised solid fuel or of atomised liquid fuel. F. L. DUFFIELD (B.P. 293,330, 31.1.27).

Motor fuel. D. COSTAGUTA (U.S.P. 1,677,273, 17.7.28. Appl., 1.6.25. Argentina, 10.3.25).—See F.P. 599,271; B., 1926, 430.

Working up acid resins obtained from the refining of mineral oil derivatives into neutral bitumens. C. SAUTERMEISTER and F. WILHELM (U.S.P. 1,677,731, 17.7.28. Appl., 19.12.25. Rumania, 30.6.25).—See F.P. 606,817; B., 1927, 182.

Separation of gaseous mixtures (B.P. 292,204). **Gas producers** (B.P. 292,052).—See I. **Road materials** (B.P. 292,871).—See IX. **Fuel for smelting** (B.P. 293,109).—See X.

III.—ORGANIC INTERMEDIATES.

Synthesis of organic compounds and ammonia from water-gas without catalysts. M. BRUTZKUS (Compt. rend., 1928, 187, 124–125).—By compressing water-gas with air up to 50 atm. as much as 9% of the carbon monoxide can be converted into organic compounds. Aldehydes and organic acids are obtained. The yield of ammonia is very small. C. W. GIBBY.

Technical analysis of carbazole. E. SCHWENK and L. WANKA (Z. anal. Chem., 1928, 74, 168–187).—A quantity of the substance containing 0.4–0.5 g. of carbazole is heated under reflux with 450 c.c. of glacial acetic acid, and 50 c.c. of a mixture of 15 pts. of chromium trioxide, 10 pts. of water, and 10 pts. of glacial acetic acid are dropped in as rapidly as the reaction will allow. The mixture is boiled for 1 hr. and distilled until 400 c.c. of acetic acid have been collected (30–45 min.). The residual liquid in the flask is diluted with 1 litre of water, treated with 15 g. of granulated zinc and 300 c.c. of 30% sodium hydroxide solution, and distilled with a vertical condenser until 300 c.c. of liquid have been collected in the receiver which has previously been charged with 50 c.c. of 0.1N-hydrochloric acid and 250 c.c. of water.

The excess of acid is then titrated as usual with methyl-red as indicator. The volume of acid neutralised by the ammonia multiplied by 0.01671 gives the weight of carbazole in the original substance. A. R. POWELL.

Determination of alcohol and ether. CHENEL.—See XXII.

PATENTS.

Manufacture of carbon disulphide. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 293,172, 26.5.27).—Carbon disulphide and hydrogen or gases containing it are produced by treating hydrocarbons (especially gaseous ones such as methane) with gaseous compounds which supply sulphur under the working conditions (especially excess of hydrogen sulphide), at high temperatures or in the electric arc, in the presence or absence of catalysts.

B. FULLMAN.

Production of methyl and other alcohols and preparation of catalysts therefor. SYNTHETIC AMMONIA & NITRATES, LTD., and R. G. FRANKLIN (B.P. 293,056, 23.12.26).—Methyl alcohol etc. is made from carbon monoxide and hydrogen under pressure using a catalyst obtained by heating a mixture of basic zinc carbonate and basic chromium carbonate (70–80 atoms of Zn to 30–20 atoms of Cr). The basic carbonates are precipitated from a mixed solution of zinc and chromium salts which may be obtained by reduction of sodium dichromate with zinc in presence of an acid, or in a dichromate cell the negative pole of which is zinc.

W. G. CAREY.

Manufacture of formaldehyde. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 293,203, 13.7.27).—The heat of the reaction products of the catalytic production of formaldehyde from methyl alcohol and air is used to preheat the constituents of the reaction mixture separately; they are mixed below the reaction temperature.

B. FULLMAN.

Manufacture of unsaturated aldehydes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 284,458, 19.1.27).—An aldehyde containing no methylene group in the α -position is condensed with an aldehyde of the type $R \cdot CH_2 \cdot CHO$ in presence of alkali and an alcohol, any water present being in amount insufficient to cause separation into layers; aldol formation is thus prevented. Benzaldehyde and propaldehyde with alcoholic potassium hydroxide at 10° give an 86% yield of α -methylcinnamaldehyde. New unsaturated aldehydes described are α -ethylcinnamaldehyde, b.p. 132–134°/14 mm., α -isopropylcinnamaldehyde, b.p. 133–135°/13 mm., 4-methoxy- α -ethylcinnamaldehyde, b.p. 169–172°/13 mm., 4-methoxy- α -n-amylicinnamaldehyde, b.p. 195–200°/13 mm., Δ^{α} - γ -ethyl- α -n-amyloctenaldehyde, b.p. 150–155°/10 mm., Δ^{α} - γ -ethyl- α -n-amyloctadienaldehyde, b.p. 150–155°/10 mm., Δ^{α} - δ -phenyl- α -ethylpentadienaldehyde [α -cinnamylidenebutaldehyde], b.p. 172–180°/12 mm., the corresponding α -methyl (m.p. 58°, b.p. 170–180°/15 mm.) and α -n-amylic (b.p. 203–210°/15 mm.) compounds, Δ^{α} - δ -phenyl- α - γ -dimethylpentadienaldehyde, m.p. 43°, b.p. 175–180°/14 mm., and Δ^{α} - δ -phenyl- γ -methyl- α -ethylpentadienaldehyde, b.p. 185–190°/13 mm. The products have a strong, pleasant odour. C. HOLLINS.

Purification of carbazole. C. J. THATCHER (U.S.P. 1,672,630, 5.6.28. Appl., 15.12.21).—Phenanthrene and

anthracene are removed from impure carbazole by treatment with warm carbon tetrachloride sufficient to dissolve the hydrocarbons but only a portion of the carbazole. To remove 10% of hydrocarbon, 800–1000 pts. of carbon tetrachloride at 50° are used for every 100 pts. of impure carbazole. C. HOLLINS.

Manufacture of paraformaldehyde. K. SATOR and W. PFANNMÜLLER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,677,730, 17.7.28. Appl., 26.6.26. Ger., 2.7.25).—See B.P. 260,908; B., 1927, 59.

1-Methyl-2:5:6-trichloro-3-aminobenzene-4-sulphonic acid. H. WAGNER and B. VOSSEN, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,677,536, 17.7.28. Appl., 13.8.27. Ger., 11.9.26).—See B.P. 277,372; B., 1928, 440.

Acetone and butyl alcohol (U.S.P. 1,672,487).—See XVIII.

IV.—DYESTUFFS.

Constitution of Hansa Yellow 3G, 5G, and 10G (M.L.B.), and Permanent Yellow R and 4R (A.G.F.A.). A. H. BURR and F. M. ROWE (J. Soc. Dyers and Col., 1928, 44, 205–207).—Hansa Yellow 3G, m.p. 250°, is 4-chloro-2-nitroaniline \rightarrow acetoacetanilide (cf. B.P. 9633 of 1910); the 5G mark is *o*-nitroaniline \rightarrow acetoacetanilide, and has m.p. 206°; the 10G mark is 4-chloro-2-nitroaniline \rightarrow acetoacetic *o*-chloroanilide (B.P. 23,791, 1911), and has m.p. 258°. Acetoacetaryl-amide couplings give much less diazo compound when treated with fuming nitric acid than do other azo couplings.

Permanent Yellow R, m.p. 263°, is *o*-chloroaniline \rightarrow 1-*p*-nitrophenyl-3-methyl-5-pyrazolone (U.S.P. 988,870), the coupling component, m.p. 222°, being made from *p*-nitrophenylhydrazine. The 4R mark, m.p. 188°, is *o*-chloroaniline \rightarrow 1-phenyl-3-methyl-5-pyrazolone.

C. HOLLINS.

Azoic colours. RATH. Effect of sulphur dioxide on azo dyes. KING.—See VI. Pigments and lakes. GREEN.—See XIII.

PATENTS.

Manufacture of thiomorpholines of the anthraquinone series. I. G. FARBENIND. A.-G. (B.P. 263,179 and Addn. B.P. 263,843, [A] 17.12.26, [B] 29.12.26. Ger., [A] 17.12.25, [B] 29.12.25. [A, B] Addn. to B.P. 263,178; B., 1928, 398).—(A) The *o*-amino- β -hydroxyalkylthiolanthraquinones of B.P. 263,178 are converted by acid condensing agents (oleum, sulphuric acid, zinc chloride) into thiomorpholine derivatives which are dyes for acetate silk. Examples are: the condensation products from 1-amino-2- β -hydroxyethylthiolanthraquinone (red), 1-amino-2- β - γ -dihydroxypropylthiolanthraquinone (red sulphonated dye), 3-chloro-1:4-diamino-2- β -hydroxyethylthiolanthraquinone (blue), 1:5-diamino-2:6-di- β -hydroxyethylthiolanthraquinone (bordeaux-red). (B) Similar morpholine derivatives are obtained by condensing 1-amino-2-thiolanthraquinone with ethylene dihalides and cyclising the product by heating, e.g., at 100° or in trichlorobenzene with addition of copper. Examples are: ethylene dibromide, chlorobromide, or dichloride, or β - γ -dichloropropyl alcohol with

1-amino-2-thiol-, 1:5-diamino-2:6-dithiol-, 1:8-diamino-2:7-dithiol-, 1-amino-4-*p*-toluidino-2-thiol-, 2-amino-3-thiol-anthraquinones; 4-bromo-1-amino-2- β -bromoethylthiolanthraquinone heated in aniline to close the thiomorpholine ring and then further with sodium acetate and copper to give the 3-anilino-compound, which by sulphonation yields a greyish-blue acid wool dye.

C. HOLLINS.

Manufacture of acid dyes of the anthraquinone series. I. G. FARBERIND. A.-G. (B.P. 263,795, 20.12.26. Ger., 23.12.25. Addn. to B.P. 263,178; B., 1928, 398).—The thiomorpholines of the prior patent, *e.g.*, those from 1-amino-2- β -hydroxyethylthiolanthraquinone, 2-chloro-1:4-diamino-3- β -hydroxyethylthiolanthraquinone, and 1:5-diamino-2:6-di- β -hydroxyethylthiolanthraquinone, are sulphonated to give acid dyes.

C. HOLLINS.

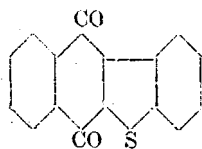
Manufacture of vat dyes of the dibenzanthrone series. I. G. FARBERIND. A.-G. (B.P. 286,602, 13.12.26. Ger., 14.12.25. Cf. B.P. 262,819; B., 1928, 399).—3:3'-Dibenzanthronyls, carrying halogen or methyl substituents but having the 4:4'-positions free, are converted by alcoholic alkali at 120–135° into corresponding substituted dibenzanthrones. 2:2'-Dimethyl-3:3'-dibenzanthronyl, prepared by oxidising the dimethylbenzanthrone from anthrone and α -methylacraldehyde, gives a blue-violet vat dye; from 9:9'-dichloro-3:3'-dibenzanthronyl a red-violet vat dye is obtained.

C. HOLLINS.

Manufacture of dyes containing chromium. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 292,344, 9.6.27).—4-Chloro-*o*-aminophenol is diazotised and coupled with a naphtholmonosulphonic acid (1:4-, 1:5-, 2:4-, or 2:6-), and the resulting dye is boiled with a chromic salt (*e.g.*, formate). [Stat. ref.]

C. HOLLINS.

Manufacture of condensation products derived from thionaphthen-2:3-dicarboxylic acid. I. G. FARBERIND. A.-G. (B.P. 261,383, 10.11.26. Ger., 10.11.25).—Thionaphthen-2:3-dicarboxylic anhydride (or derivative) is condensed with an aromatic compound in presence of aluminium chloride, and the resulting *o*-carboxyketone is cyclised with sulphuric acid, or, after conversion into the acid chloride, with aluminium chloride, to form analogues of naphthanthraquinone containing a thiophen nucleus. From these by condensation with glycerol to a benzanthrone analogue and subsequent alkaline fusion analogues of dibenzanthrone are obtained. Thionaphthen-2:3-dicarboxylic anhydride with benzene and aluminium chloride gives 3- (or 2-)benzoylthionaphthen-2 (or 3)-carboxylic acid, m.p. 216°, which is converted into the acid chloride and thence into 2:3-phthaloylthionaphthen (annexed formula), m.p. 212–213°, a yellow vat dye for wool. From chlorobenzene an *o*-carboxyketone, m.p. 198–199°, and a chloro-2:3-phthaloylthionaphthen, m.p. 215–220°, are obtained. 5-Chloro-7-methylthionaphthen-2:3-



dicarboxylic acid, m.p. 259–260°, gives an anhydride, m.p. 198–199°, from which are similarly

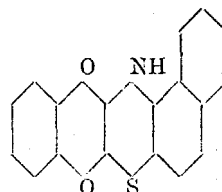
obtained an intermediate *o*-carboxyketone, m.p. 256–257°, and 5-chloro-2:3-phthaloyl-7-methylthionaphthen, m.p. 278°. Cyclisation of benzoylthionaphthencarboxylic acid with 20% oleum gives a sulphonated product, which is a yellow acid dye. The "benzanthrone," m.p. 182°, from 2:3-phthaloylthionaphthen is converted by alkaline fusion into a blue vat dye for cotton.

C. HOLLINS.

Manufacture of 2:3-phthaloylthionaphthen and derivatives thereof. I. G. FARBERIND. A.-G. (B.P. 265,193, 25.1.27. Ger., 29.1.26).—Thionaphthens are condensed with phthalic anhydride (or derivatives) in presence of aluminium chloride, and the resulting carboxyketone is cyclised in the usual manner. 3-*o*-Carboxybenzoylthionaphthen, from phthalic anhydride and thionaphthen, is converted by way of the acid chloride into 2:3-phthaloylthionaphthen, m.p. 212–214° (cf. B.P. 261,383, preceding).

C. HOLLINS.

Manufacture of carbazolequinones. I. G. FARBERIND. A.-G. (B.P. 264,530, 15.1.27. Ger., 15.1.26).—Sulphur is removed from thiazinequinones, *e.g.*, by heating with copper powder, with production of corresponding carbazolequinones.



Thus, pheno- $\beta\beta$ -naphthathiazine-5:10-quinone when heated with copper in a stream of carbon dioxide yields $\beta\beta$ -naphthacarbazole-5:11-quinone, m.p. 307°. $\alpha\beta\beta'$ -Dinaphthathiazine-8:13-quinone (annexed formula) when heated with copper and naphthalene yields $\alpha\beta\beta'$ -dinaphthacarbazole-7:12-quinone, an orange-red vat dye.

C. HOLLINS.

Halogen derivative of the benzanthrone series. O. BRAUNSDORF, E. HOLZAPFEL, and P. NAWIASKY, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,677,545, 17.7.28. Appl., 23.3.26. Ger., 27.3.25).—See B.P. 249,890; B., 1927, 647.

Solid diazo salts. F. KELLER and K. SCHNITZSPAHN, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,677,558 and 1,677,640, 17.7.28. Appl., 11.2.27. Ger., 12.2.26).—See B.P. 265,985; B., 1928, 328.

Metal compound of azo dyestuff. F. STRAUB, H. SCHNEIDER, and J. SPIELER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,677,534, 17.7.28. Appl., 7.4.25. Switz., 20.4.24).—See B.P. 232,620; B., 1925, 954.

Converting dyes into solution etc. (B.P. 264,860).—Sec XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

X-Ray investigation of woollen samples. T. D. THREADGOLD (J. Text. Inst., 1928, 19, 233–236 T).—It is highly improbable that the wool fibre is truly amorphous; it would appear to consist of an aggregate of extremely small crystalline particles, *i.e.*, it is colloidal.

B. P. RIDGE.

Thread-like structure of natural silk. P. P. VON WEIMARN (Kolloid-Z., 1928, 45, 162–164).—See B., 1928, 562.

Cellulose resources. II. Cellulose from field crops. G. M. ROMMEL (Ind. Eng. Chem., 1928, 20, 587—591; cf. B., 1928, 477).—A summary of the records of experimental stations of various states as to the yields of corn stover and grain per acre with and without soil treatment, the average yield of stalks, and the maximum annual production of dry material obtainable under different conditions of planting. The possibility of employing these and other field crops as sources of cellulose is discussed. F. R. ENNOS.

***Helianthus argophyllus*. Commercial possibilities as indicated by its composition.** K. S. MARKLEY and W. T. SCHREIBER (Ind. Eng. Chem., 1928, 20, 636—637).—The cellulose content (46.5%) of the stalks of *Helianthus argophyllus*, the white-leaved or Texas sunflower, is considerably below that of spruce wood (60%), but compares more favourably with that of cotton stalks (45—49%) and bagasse (50—55%); it may, however, be reckoned as a possible source of cellulose. F. R. ENNOS.

Utilisation of Moroccan woods. G. DUPONT and M. SOUM (Bull. inst. pin, 1927, No. 33, 41—44).—Wood of *Cedrus atlantica* contains (dry) total cellulose 55.6 (α -cellulose 40.9), lignin 27.10%, and yields when heated under pressure with sodium hydroxide solution 48.7% of easily bleached pulp, having fibres $2.9\text{--}4.5 \times 0.015\text{--}0.030$ mm. Sandarac (*Callitris quadrivalis*) wood is unsuitable for pulping. Portuguese *Eucalyptus globulus* wood contains (dry) ash 0.29, pentosans 4.37, methylpentosans 2.57, lignin 12.66, α -cellulose 50.01, β -cellulose 5.83, γ -cellulose 26.18%, and yields 48.5% of easily bleached pulp.

CHEMICAL ABSTRACTS.

Wood. I. Analysis of wood rays in two hardwoods. W. M. HARLOW and L. E. WISE (Ind. Eng. Chem., 1928, 20, 720—722).—In the two species of hardwood examined, white oak (*Quercus alba*, L.) and Australian flame she-oak (*Casuarina inophloia*), the wood rays are large, and can be removed in amounts sufficient for analysis. The lignin content is appreciably higher than that of the total wood, whilst the cellulose content is lower. The flame she-oak rays had alcohol-benzene-soluble 20.2, lignin 36.7, pentosans 11.6, and pentosan-free cellulose 26.5%, whilst the white oak rays had water-soluble 7.5, lignin 32.7, pentosans, 28.5, and pentosan-free cellulose 28.2%.

W. J. POWELL.

Occurrence of pinitol [pentahydroxymethoxycyclohexane] in redwood. E. C. SHERRARD and E. F. KURTH (Ind. Eng. Chem., 1928, 20, 722—723).—Pentahydroxymethoxycyclohexane was obtained from redwood heartwood (*Sequoia sempervirens*) by extracting the air-dried material with water and concentrating the solution. After purification it has m.p. 185° , $[\alpha]_D^{25} + 65.4^\circ$; it does not reduce Fehling's solution, and has the same degree of sweetness as sucrose. In addition, a new cyclose (sesquioxide), $C_8H_{16}O_7$, m.p. 234° , was obtained from the aqueous extract; it sublimes with little decomposition, and the tentative formula $C_6H_5(OH)_6 \cdot CH_2 \cdot OMe$ is suggested. W. J. POWELL.

Degree of swelling of hydrated cellulose [artificial silk]. O. FAUST (Cellulosechem., 1928, 9, 74—

75).—Changes in length of artificial silk fibres on immersion in water and in sodium hydroxide solutions show that the degree of swelling decreases with increasing temperature, the difference being greater the higher the concentration of sodium hydroxide. The degree of swelling, as determined by length measurements, is lower in sulphuric acid solutions than in water.

W. J. POWELL.

Use of starch in paper. H. WREDE (Papier-Fabr., 1928, 26, 301—309).—Comparison is made between the properties of rice, wheat, maize, and potato starches from the point of view of sizing in paper manufacture, and a method is described for determining the viscosity and adhesive power of starch pastes. Starch is useful not only for improving the sizing and the quality of paper but also as a filling and finishing material. Wheat starch is absorbed by the fibres to the greatest extent, but maize is to be preferred on account of its greater gel formation and cheapness. Temperatures of pasting differ for the various starches, but in sizing, instead of partial pasting, thorough cooking is recommended, with application of the paste to the fibres warm. The addition of nekal BX increases the absorption of starch by the fibres and assists dyeing, whilst for papers resistant to moisture and rain, such as poster papers, montan wax should be used. Starch gives greater uniformity and closeness of surface texture, and a mixture of starch and water-glass is specially useful in this respect. The uses of starch, raw, pasted under different conditions (e.g., by means of sodium hydroxide, or by cooking), and in combination with such substances as rosin, sodium silicate, nekal, and montan wax, and the effects produced in the finished material, are described for writing, printing, parchment, and other papers. B. P. RIDGE.

Nitrocellulose lacquers. HOFMANN and REID.—See XIII. **Carbon in nitrocellulose.** CARRIÈRE and GUIBERT. **Camphor and nitrocellulose.** DESMAROUX.—See XXII.

PATENTS.

Extracting and purifying the fatty materials contained in waste waters from the washing of wool. C. VAN OVERSTRAETEN (B.P. 275,627, 3.8.27. Belg., 6.8.26).—The precipitated fatty matter is washed with warm water and boiled for about 1 hr. with 0.5—2% of sodium carbonate (preferably without addition of any further quantity of water) to saponify fatty acids and coagulate albuminous matter. The resulting mixture is dried and heated, preferably under pressure, at 105° for $\frac{3}{4}$ hr. The neutral fat which separates during this operation is decanted, washed with boiling water, and dried. D. J. NORMAN.

Manufacture of hair felt. A. L. CLAPP, Assr. to BECKWITH MANUF. CO. (U.S.P. 1,674,945, 26.6.28. Appl., 12.9.23).—Cellulosic material is beaten with a soap having the slimy characteristics of a straight-chain fatty acid soap; long hair is then added to the beater and the beating action continued without cutting until the fibres are thoroughly separated. The resulting stock is run on a paper machine. D. J. NORMAN.

Treatment of textile materials. Production of cellulosic fabric. TOOTAL BROADHURST LEE CO., LTD.,

and [A, B] R. P. FOULDS, J. T. MARSH, F. C. WOOD, [A] H. BOFFEY and J. TANKARD (B.P. 291,473—4, 1.12.26 and [A] 26.3.27, [B] 7.6.27).—Cellulosic and other creasable fabrics are rendered less susceptible to creasing and crumpling when crushed or folded under pressure, whilst retaining their original suppleness, if they are impregnated with (B) phenol-formaldehyde resins, or (A) other synthetic resins. The final condensation of the resin should be effected on the fabric itself, and the catalyst and the temperature should be so chosen that undue tendering of the fabric is avoided. In some cases the alkali left after mercerisation may serve as the condensing agent. *E.g.*, medium-weight plain cotton cloth is passed through caustic soda solution ($d > 1.10$) and then, after squeezing, into a reaction mixture containing equal parts by wt. of phenol and 40% formaldehyde solution. The impregnated fabric is squeezed, dried at a temperature not exceeding 60°, and further treated with hot or cold 40% formaldehyde solution for varying periods according to the finish required, after which it is dried without washing and heated at, *e.g.*, 180° for 2–5 min. Excess of reagents is finally removed by soap boiling. D. J. NORMAN.

Manufacture of fabrics resisting the disaggregating action of "Yperite" or like gases. G. CARDILE, and INDUSTRIA ARTIFICI CAOUTCHOUC (I.A.C.) (B.P. 292,813, 23.9.27).—The fabric is coated with a mixture of animal glue and glycerin and treated with, *e.g.*, formaldehyde to render the glue insoluble. A thin coating of rubber or other moisture-resisting material is then applied and vulcanised.

D. J. NORMAN.

Cleaning processes. A. E. HATFIELD, and ACHILLE SERRE, LTD. (B.P. 289,581, 15.2.27. Addn. to B.P. 266,850; B., 1927, 579).—The process of the prior patent is applied to the soiled liquids resulting from ordinary laundering operations. D. J. NORMAN.

Composition of matter [cellulose impregnated with sulphur] and process of forming the same. H. A. GILL. From F. N. BURT Co., LTD. (B.P. 291,500, 28.2.27).—Fibrous material may be impregnated with sulphur so that the final composition contains 65–80% S if substances such as naphthalene or its derivatives, diphenyl, carbazole, triphenyl phosphate, or similar compounds containing more than one cyclic nucleus in the molecule are added to the molten sulphur to increase its penetrability and to confer on it the property of wetting cellulose fibres. Satisfactory results are obtained by using as an impregnating medium under atmospheric pressure molten sulphur containing 5% of naphthalene at 130–135°. The resulting products are hard and capable of being machined, and show good insulating properties. D. J. NORMAN.

Treatment after spinning of artificial silk made from viscose. DEUTS. ZELLSTOFF-TEXTILWERKE, G.M.B.H., Assees. of SPINNSTOFFFABR. ZEHLENDORF G.M.B.H. (B.P. 268,783, 29.3.27. Ger., 30.3.26).—The filaments leaving the spinning bath are collected on a foraminated spool made from a material, *e.g.*, stainless steel (V2A steel), which resists the action of the desulphurising agent, and are washed, desulphurised,

and bleached thereon, the various treating solutions being made to flow through the cake and the foraminated spool by differential pressure. Alternatively, the silk may be collected on a spinning spool and transferred to a foraminated twisting spool for desulphurisation, bleaching, etc. D. J. NORMAN.

Manufacture of artificial textile threads, fibres, filaments, etc. COMPT. DES TEXTILES ARTIF. SOC. ANON., and H. L. J. CHAVASSIEU (B.P. 268,734, 10.2.27. Fr., 3.4.26).—Artificial fibres closely resembling wool are made by incorporating with viscose a quantity of finely divided sulphur equal to or greater than that found in natural wool. This is effected by adding a mixture of 1 mol. of sodium thiosulphate and 2 mols. of sodium sulphide either to the spinning bath in sufficient quantity to give 6–10 g./litre of colloidal sulphur, or to the spinning solution, or by immersing coagulated viscose filaments for about 15 min. at the ordinary temperature in a bath containing per litre 25–75 g. of caustic soda, 80–150 g. of sodium sulphide, and 120–200 g. of sodium thiosulphate, and then transferring the impregnated filaments, after draining, to a tepid bath containing 100–200 g. of sulphuric acid monohydrate per litre. The wool-like character of the product is enhanced if albuminoids are added to the spinning solution as described in F.P. 395,402. D. J. NORMAN.

Treatment of threads of artificial silk and other materials to make them act like wool. E. K. SCOTT (B.P. 291,120, 24.11.26).—Fulling and felting properties can be imparted to artificial fibres by crimping and roughening the filaments either during the spinning operation, *e.g.*, by spinning through a nozzle set tangentially and angularly in the wall of a horizontal tube through which is forced a coagulating liquid carrying in suspension a coarse powder, or while the filaments are still in a plastic condition by the use of, *e.g.*, finely-scored fluting rollers. D. J. NORMAN.

Manufacture of soluble cellulose esters of higher fatty acids. I. G. FARBERNID. A.-G. (B.P. 283,181, 6.1.28. Ger., 6.1.27. Cf. B.P. 201,510; B., 1924, 128).—Cellulose esters of the higher fatty acids are prepared by heating untreated cellulose and a higher fatty acid chloride in the presence of a base (*e.g.*, pyridine, quinoline) optionally with addition of a diluent which is a solvent for the required ester, at 100–140° (there being a temperature limit for each ester below which the product is insoluble), and precipitating the reaction product by the addition of alcohol, acetone, or the like.

D. J. NORMAN.

Production of partially hydrolysed cellulose acetates and of products such as threads, films, lacquers, etc. therefrom. N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (B.P. 292,398, 8.10.27. Holl., 20.7.27).—The partial hydrolysis of primary cellulose acetates may be effected without degrading the cellulose molecule and with the production of high-viscosity cellulose acetates by operating at 20° with dilute sulphuric acid and prolonging the hydrolysis until the viscosity of an acetone solution of the product has, after falling to a minimum value, risen again to approximately $1\frac{1}{2}$ times the minimum value. Thus, in one case after hydrolysis for 72, 96, 120, and 144 hrs., the viscosity

values of a 15% solution of the product in acetone were 240, 150, 170, and 260, respectively. D. J. NORMAN.

Production of cellulose articles from waste tanbark or waste bark of Coniferæ and foliage trees. C. ZARFEL and W. D. STROEVER (B.P. 289,676, 14.7.27).—The material, optionally in admixture with pulp fibres, is boiled for 2—3 hrs. in a closed vessel with 3—4% caustic soda solution and is then washed, ground, and converted into products suitable for use, *e.g.*, as insulating materials, or stuffing for steam or water fittings. D. J. NORMAN.

Preparation of cellulose. P. J. A. MAIGNEN (U.S.P. 1,672,895, 12.6.28. Appl., 2.7.24).—Cellulose suitable for papermaking is separated from the non-cellulose of plant tissue without impairing the value of the non-cellulosic constituents for plant fertilising purposes by subjecting the raw material under atmospheric pressure to the action of caustic soda solution progressively generated in the cooking vessel from a mixture of sodium carbonate and calcium hydroxide. D. J. NORMAN.

Softening paper. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 292,430, 16.3.27).—The paper is impregnated with sorbitol or its dehydration products or derivatives either alone or in admixture with other softening agents (*e.g.*, glycerol). D. J. NORMAN.

Manufacture of insulating paper, paste-board, card-board, and moulded articles formed from fibrous masses. H. FRIEDLÄNDER (B.P. 262,828, 14.12.26. Ger., 14.12.25).—Saponified or emulsified hard waxes, *e.g.*, montan wax, either alone or in admixture with other hard waxes or their by-products or natural or artificial resins, are mixed with fibrous pulp in quantities approximating to 35—50% on the weight of dry pulp and are then precipitated by the addition of, *e.g.*, alum. The pulp-wax mixture is then formed into sheets or moulded. The resulting products, which remain pliable despite their content of hard waxes, may be glazed, or impregnated with further quantities of hard wax as described in B.P. 236,224 (B., 1926, 986). D. J. NORMAN.

Pulping of wood. F. G. RAWLING (U.S.P. 1,673,089, 12.6.28. Appl., 1.9.26. Can., 19.3.27).—Wood chips are digested at 100—125° with an aqueous solution of a normal sulphite and a salt of a weakly ionised polybasic acid. The liquor is then removed and the chips are heated at a higher temperature. D. J. NORMAN.

Manufacture of resin-coated board. G. M. CLARK. From AGASOTE MILLBOARD Co. (B.P. 291,633, 12.7.27).—A layer of artificial fusible resin, preferably in admixture with fillers, is applied to a fibrous spongy base containing a fusible binder with a low coefficient of expansion, *e.g.*, asphalt, and the whole is compacted and the resin rendered infusible by heat and pressure. D. J. NORMAN.

[Pumping] apparatus for use in manufacture of artificial silk etc. BRIT. CELANESE, LTD., and E. KINSELLA (B.P. 293,325, 23.12.26 and 22.10.27).

Manufacture [twisting and winding] of artificial silk or like threads and apparatus therefor. BRIT. CELANESE, LTD., and S. A. WELCH (B.P. 293,371, 4.4.27).

Spinning boxes used in manufacture of artificial silk and like material. BRIT. THOMSON-HOUSTON Co., LTD., A. P. YOUNG, H. W. H. WARREN, and R. J. CHAPMAN (B.P. 293,060, 22.1.27).

Spindles of spinning machines for artificial silk etc. HARBENS (VISCOSILK MANUFACTURING), LTD., and W. E. SHARPLES (B.P. 293,596, 3.9.27).

Recovery of [paper pulp] fibres from liquids. A. M. R. KARLSTRÖM (B.P. 277,310, 17.8.27. Swed., 7.9.26).

Manufacture and use of carbon papers and like transfer materials. FELIX, PELTZER & Co. (B.P. 279,375, 8.3.27. Ger., 23.10.26).

Manufacture of textile fibres from animal hair and bristles. H. SCHWEITZER, Assr. to AMER. LANIL CORP. (U.S.P. 1,677,149, 17.7.28. Appl., 17.11.22. Ger., 13.2.22).—See G.P. 382,085; B., 1924, 90.

Clarification of benzene (B.P. 292,433).—See II. **Regeneration of rubber** (B.P. 270,675).—See XIV. **Imitation wash leather** (B.P. 289,592).—See XV.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Azoic [Naphthol AS] colours. E. J. RATH (J. Soc. Dyers and Col., 1928, 44, 10—14).—The difficulties encountered in the preparation of the impregnating baths for members of the Naphthol AS series, owing to decomposition of the sodium naphthoxide on the fibre, are probably due mainly to hydrolysis (*cf.* Higgins, B., 1927, 649), which is accelerated by carbon dioxide. Differences in the stability of the different naphthols appear to be due to differences in the arylide residue. The decomposition may be prevented by using a large excess of sodium hydroxide or by enhancing the acid nature of the naphthols by the introduction of acid groups. In practice, the former method is not feasible. Addition of formaldehyde to the naphthol solutions yields additive compounds, the more acidic nature of which offers greater resistance to hydrolysis. (The formaldehyde is split off when the additive compound couples with a diazo compound.) The resistance to air develops some time (*e.g.*, 5 hrs.) after the addition of formaldehyde to the impregnating bath. Arranged according to the time required for complete stability to air after addition of formaldehyde the order is Naphthol AS-BS, AS-BG, AS, AS-BO, AS-TR, AS-SW, and AS-RL. The addition of formaldehyde to Naphthol AS-BR weakens the colouring power. Raising the temperature tends to cause precipitation of the naphthol. Addition of formaldehyde also increases the colour intensity of the impregnation baths, the degree of dispersion of the colloidal naphthol being diminished, and increases the substantivity of the naphthols, thereby giving deeper shades. Substantivity is more pronounced at low temperatures and varies inversely as the concentration. For each naphthol there is a maximum value of naphthol absorbed substantively, and beyond this point the mechanical absorption alone increases, the fastness to rubbing therefore decreasing at the same time. The smaller the ratio of cotton to liquor, the greater is the absorption of naphthol obtained. Determinations of the substantivity of the various

Naphthol AS products in different ratios of goods to liquor show that the substantivity increases in the order Naphthol AS, AS-D, AS-RL, AS-BG, AS-BS, AS-TR, AS-BO, AS-SW, to AS-BR. The additive compounds of pyridine and Naphthol AS (Higgins, *loc. cit.*) could not be obtained. R. BRIGHTMAN.

Effect of sulphur dioxide on azo dyestuffs and a proposed standard test for fastness to stoving. A. T. KING (J. Soc. Dyers and Col., 1928, 44, 14—18).—The maximum reactivity of azo dyes with sulphur dioxide in presence of sodium hydroxide previously described (A., 1927, 1180) may be realised fortuitously in practice, even with dyestuffs of reputedly good fastness to stoving. Appreciable amounts of the azo sulphite may be formed in stoving if the sulphur dioxide is too dilute or small in quantity, or if the material is very alkaline. Accidental exposure of wool or yarn-dyed material to sulphur dioxide may also produce fading, the active range being dependent rather on the relative amounts of alkali and sulphur dioxide than on the actual quantity of the latter. Cotton goods dyed with direct azo dyes are specially liable to this fault. Residual sulphite from previous processes may also cause trouble, and migration of alkali (B., 1927, 873) is a contributory factor in sulphur dioxide faults. The present standard stoving test is criticised adversely, and the following alternative test, which covers the "active range," is suggested: The pattern is brought to a definite alkalinity by the use of bromothymol-blue indicator cloth (B., 1927, 873) in the same conditions, and then divided into two parts. These, with the indicator cloth, are hung in the stoving vessel, one portion being removed immediately the indicator cloth turns just yellow. This is kept damp under a bell-jar over water and examined along with the other portion at the end of the stoving period. The fastness is expressed in the present range of five or better in a range of three grades. R. BRIGHTMAN.

Modified stoving test [for dyed wool]. F. L. GOODALL (J. Soc. Dyers and Col., 1928, 44, 145—146). A. T. KING (*Ibid.*, 146—147).—The fastness to stoving (treatment with sulphur dioxide) of coloured wool materials is determined by spotting the material with solutions containing (1) 5% of sodium sulphite, (2) 5% of sodium bisulphite and 0.4 mol.-equiv. of caustic soda, (3) 5% of sodium bisulphite, and (4) 5% of sodium bisulphite and 0.5 mol.-equiv. of sulphuric acid, and observing the change of shade thereby produced at the end of 12 hrs. This test, using solution (2), corresponds to the conditions specified in the King test (cf. preceding abstract), but is considerably less laborious and arbitrary; further, it has yielded results which agree with experience obtained in large-scale stoving processes. The fastness to stoving of many wool dyes as determined by the new test differs from that indicated by the standard stoving test now in use. Dyes made from α -naphthol are much more resistant to the new test than those derived from β -naphthol. Thus the yellower members of the Polar Red dyes are derivatives of β -naphthol and are more sensitive to stoving than the bluer members which are α -naphthol derivatives. Many direct dyeings on wool of chrome colours which are sensitive to stoving are resistant after chroming, thus

indicating the formation of chrome lakes on the wool fibre.

KING prefers his original test, since it simulates more closely the actual conditions of stoving and avoids the use of excessive amounts of a sulphite or bisulphite. The greater resistance to stoving shown by α -naphthol dyes is ascribed not to the configuration of the α -naphthol nucleus but to the greater inhibiting effect of the substituent groups when introduced into the α -naphthol nucleus. A. J. HALL.

Elastic properties of starch film. PEIRCE.—See XVII.

PATENTS.

Dyeing, printing, or stencilling of materials made of or containing cellulose derivatives. H. DREYFUS (B.P. 292,180—1, 292,452—3, 14.12.26).—Acetate silk and other cellulose esters and ethers, including immunised cotton, are dyed with coloured compounds characterised by: (A) azo groups and hydroxylated alkylamino-groups, *e.g.*, *p*-(β - γ -dihydroxybutylamino)benzeneazo- α -naphthylamine (golden); picramic acid \rightarrow β - γ -dihydroxypropylaniline (yellowish-brown); 4-nitro-2-amino- β - γ -dihydroxypropylaniline \rightarrow *p*-xylylene (yellow); (B) an ω -amino- or alkylated amino-group and no azo group, *e.g.*, 1-methylamino-4-aminomethylantraquinone (by reduction of 1-methylamino-4-cyanoanthraquinone; red); 1-methylamino-acetamido-4-methylaminoanthraquinone (red-violet); 1- β - γ -dihydroxypropylaminoacetamido-4- β - γ -dihydroxypropylaminoanthraquinone (red-violet); *N*-(γ -amino- β -hydroxypropyl)pyrimidoneanthrone (from pyrimidone-anthrone and β -chloro- β -aminoisopropyl alcohol; yellow); 2-nitro-4 : 4'-bis- β -aminoethylaminodiphenylmethane (pale yellow); (C) an azo group, or a nitro-diarylamino-group, or a nitrodiarylmethane, or a pyridoneanthrone group, containing a nuclear urethane residue, *e.g.*, 2 : 4-dinitroaniline \rightarrow *o*-hydroxyphenylurethane (orange); 5-amino-*m*-anisylurethane \rightarrow aniline (developed on the fibre with *o*-hydroxyphenylurethane; orange); 4 : 4'-dinitrodiphenylurethane (yellow); 2-nitro-4 : 4'-di(carbophenoxyamino)diphenylmethane (pale yellow); 1-carbomethoxymethylaminopyridone-anthrone (from chloropyridoneanthrone and *N*-methylmethylurethane, yellow); (D) an azo group, with one or more ω -amino-groups in the diazo component, *e.g.*, *p*- β -aminoethoxybenzeneazo- α -naphthylamine (various shades by development); *o*-aminophenylethylamine \rightarrow β -aminoethylaniline (brownish-yellow). All the dyes may be used with solubilising agents *etc.* if desired. C. HOLLINS.

Dyeing, printing, and stencilling of materials made of or containing cellulose derivatives. H. DREYFUS (B.P. 291,816, 14.12.26. Addn. to B.P. 291,118; B., 1928, 601).—Thiourethanes, corresponding with the thioureido-derivatives claimed in the prior patent, are used for dyeing cellulose esters or ethers. Examples are: 4-acetamido-1-antraquinonylthiourethane, $\text{NHAc} \cdot \text{C}_{14}\text{H}_6\text{O}_2 \cdot \text{NH} \cdot \text{CS} \cdot \text{OEt}$ (orange) and aniline \rightarrow α -naphthylamine \rightarrow 3-hydroxy-4-carboxyphenylthiourethane (golden-orange). C. HOLLINS.

Coloration of cellulose esters and ethers. Soc. CHIM. DES USINES DU RHÔNE (B.P. 275,553, 25.2.27.

Fr., 3.8.26).—The reagents required for the production of mineral pigments by simple reactions or double decomposition are introduced in aqueous solution into a solution of the cellulose ester or ether in an organic solvent, and the coloured product is precipitated by, *e.g.*, the addition of water. Preferably each reagent is added to a separate quantity of the ester or ether solution and the whole subsequently mixed with vigorous agitation. *E.g.*, a concentrated aqueous solution containing 5 g. of ferric chloride is thoroughly mixed with a solution of 100 g. of cellulose acetate in 500 g. of acetone; an aqueous solution containing 6.5 g. of potassium ferrocyanide is then stirred in, and, when the reaction is complete, the ester, containing Prussian Blue in an almost colloidal state, is precipitated with water, washed free from reaction by-products, and dried.

D. J. NORMAN.

[Circulating device for] apparatus for dyeing hanks of yarn. P. F. HÖLTZING (B.P. 280,844, 9.3.27. Ger., 19.11.26).

Coloured rubber (B.P. 277,034).—See XIV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Industrial future of the fixation of nitrogen as oxide by the electric discharge. E. BRINER (Bull. Soc. chim. Belg., 1928, 37, 169—185).—The present thermal efficiency of the best arc furnaces in terms of the heat requirements of the reaction $N_2 + O_2 = 2NO$ —43,200 cal. does not exceed 3%. The product of a kw.-year is 120—140 kg. of fixed nitrogen. With the Haber process, using electrolytic hydrogen, it is 600 kg. (as ammonia). If the ammonia is afterwards oxidised to nitric acid the thermal efficiency of the whole operation is about 21%. An improvement in the thermal efficiency of the arc process might, therefore, once more render it the more economic. From the purely thermal point of view it is improbable that such a result would be obtained merely by increase of arc temperatures, as a maximum nitric oxide concentration would probably be reached at about 3500°. The dissociation of molecules into atoms at above 2000° must be allowed for in this connexion. The author and others have, however, obtained yields of as much as 12—14.5% at the optimum pressure of 100 mm. This optimum also applies to the synthesis of ammonia by an electric spark, and in the latter case the optimum gas concentrations are not those demanded by the chemical equation. It is therefore concluded that ordinary thermal relations do not govern such reactions alone, but that purely electrical and photochemical effects must be considered. The number of variously charged ions which may be produced in the arc discharge is considerable, and nothing is known of the parts that they may play. Neither is anything known of photochemical effects except that radiation exercises a dissociating influence on oxides of nitrogen. That electrical effects may be of real significance is argued by analogy from an ozone apparatus which worked with an energy efficiency of 30%.

C. IRWIN.

Determination of water in bleaching powder, jelly, etc. T. SOMIYA (J. Soc. Chem. Ind. Japan,

1928, 31, 213—216).—The apparatus devised by Dean and Stark (B., 1920, 438 A) and by Kafuku is modified by attaching a three-way cock and a siphon to the bottom of the graduated tube to return the solvent automatically to the flask. The water content of bleaching powder and millet jelly have been determined using solvents heavier than water, such as carbon tetrachloride etc. It is claimed that in accuracy it compares favourably with other known methods, and is simpler and quicker in use. Y. TOMODA.

Molybdenum-blue, its properties and composition. L. A. MUNRO (Proc. Nova Scotian Inst. Sci., 1927, 16, 9—24).—Molybdenum-blue is the blue coloration obtained when molybdic acid is reduced in acid solution, the best method being the action of acid stannous chloride on ammonium molybdate solution. By the addition of excess of stannous chloride, a green and then brown solution can be obtained due to further (reversible) reduction. Molybdenum-blue forms a (negative) colloidal solution which can be bleached by animal charcoal, partly by adsorption and partly by oxidation. It can also be obtained in the crystalloidal form by dialysing the colloidal solution, precipitating the hydrogel with ammonium chloride, and evaporating the remaining solution. The formula by analysis of the hydrogel is approximately $Mo_3O_8 \cdot H_2O$, but since it is readily soluble in alcohol the water is probably not combined as hydrate (*cf.* Zsigmondy, "Chemistry of Colloids," 1917), hence the true formula is Mo_3O_8 . The formation of the blue by reduction of ammonium molybdate with stannous chloride can be used as a test for tin, and is sensitive to 1 pt. in 1,423,000 pts. of tin. S. J. GREGG.

Solubility of lubricating oil in liquid carbon dioxide. E. L. QUINN (Ind. Eng. Chem., 1928, 20, 735—737).—Solid carbon dioxide made by expanding the liquid from an ordinary commercial carbon dioxide cylinder is nearly always contaminated with lubricating oil arising from the compressor. Using a procedure similar to that described earlier (*cf.* A., 1928, 470), the maximum solubility of lubricating oil in liquid carbon dioxide (about 0.9%) was found to occur at 10°, at which temperature the components have the same density. The presence of oil does not appreciably affect the density of liquid carbon dioxide at ordinary room temperatures. The solubility of glycerol (a possible alternative lubricant) in liquid carbon dioxide is less than 0.05%. S. S. WOOLF.

Ammonia from water-gas. BRUTZKUS.—See III.
Sulphur-lime dry mix spray. GINSBURG.—See XVI.
Colloidal silver. KOLTHOFF.—See XX.
Organic matter in mixed acid. CARRIÈRE and GUBERT.—See XXII.

PATENTS.

Production of hydrocyanic acid from formamide. SYNTHETIC AMMONIA & NITRATES, LTD., and T. EWAN (B.P. 292,749, 21.5.27).—Formamide is heated in presence of a catalyst, and the optimum temperature and time of contact are produced and maintained by means of internal electrically-heated gauzes of copper, brass, or phosphor-bronze, upon which is supported a

coating of catalytic material such as refractory oxides. External heating may supplement the internal heating.
W. G. CAREY.

Apparatus for the synthetic production of ammonia from the elements. I. W. CEDERBERG, Assr. to PATENT VERWERTUNGS A.-G. "ALPINA" (U.S.P. 1,673,966, 19.6.28. Appl., 22.9.24. Nor., 28.9.23).—The entire walls of the reaction chamber are permeable and there is a space between the latter and its casing. The heat supply can be controlled.
F. G. CLARKE.

Production of ammonia-air mixtures. H. PAULING (B.P. 292,830, 31.10.27).—The ammonia is absorbed by a circulating liquid, preferably water, and is removed from solution by an air current blown through the liquid, both processes being performed in counter-current apparatus. The control of the circulation of liquid and of the amount of air is made automatic, the changes of temperature due to variations in the amount of ammonia supplied and/or variations in the sp. gr. of the solutions being utilised to operate control members.
W. G. CAREY.

Recovery of sodium bicarbonate from brines. W. A. KUHNERT (U.S.P. 1,674,474, 19.6.28. Appl., 29.3.27).—Carbon dioxide is passed into brine containing sodium carbonate at such a rate that the temperature remains below 45° and sodium sesquicarbonate crystallises; by continuing the flow of gas a further quantity of the soda is precipitated as bicarbonate and the sesquicarbonate is converted into bicarbonate. The total bicarbonate precipitate is then separated from the brine.
W. G. CAREY.

Production of alkali nitrates. W. WILD and C. BECK, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,674,077, 19.6.28. Appl., 24.9.27. Ger., 6.9.26).—Solutions of alkali chlorides contaminated with alkali sulphates are treated with nitric acid and with the oxide, carbonate, or nitrate of a metal the sulphate of which is insoluble in water.
A. R. POWELL.

Manufacture of sodium hydrosulphide. H. HOWARD, Assr. to GRASSELLI CHEM. CO. (U.S.P. 1,675,491, 3.7.28. Appl., 20.7.25).—An alkali sulphide is dissolved in a solution of an alkali hydrosulphide, and the solution is treated with sodium bicarbonate.
H. ROYAL-DAWSON.

Recovery of soluble salts from natural brines etc. C. E. DOLBEAR (U.S.P. 1,673,969, 19.6.28. Appl., 3.5.27).—A solid mixture containing borax, sodium carbonate, and a potassium salt is leached, in the presence of lime, with a hot solution containing sodium borate, chloride, and sulphate and a potassium salt.
F. G. CLARKE.

Production of alumina. C. McC. BROWN (U.S.P. 1,675,157, 26.6.28. Appl., 17.3.26).—Aluminous material containing a portion of its aluminium as sulphate is calcined at a temperature sufficient to decompose this compound, and the product is heated with ammonium sulphate, leached with water to form ammonium alum, and the aluminium precipitated as hydroxide by the ammonia released in the heat-treatment, ammonium sulphate being formed.
W. G. CAREY.

Drying of hydrogels. E. B. MILLER, Assr. to SILICA GEL CORP. (U.S.P. 1,674,558, 19.6.28. Appl., 17.10.25).—The hydrogel is partially dehydrated while stationary at a low temperature, and when the water content is so reduced that the hydrogel can withstand agitation it is heated at a considerably higher temperature while being agitated.
W. G. CAREY.

Manufacture of [silica] gels. E. B. MILLER and G. C. CONNOLLY, Assrs. to SILICA GEL CORP. (U.S.P. 1,672,768, 5.6.28. Appl., 6.5.27).—Silica gel "fines" are incorporated, with vigorous agitation, in a solution of acid and silicate of such concentration that it sets without precipitation to a hydrogel; this is then almost completely dehydrated at 75–120°, and finally at 200–300°.
C. HOLLINS.

Catalyst for the production of hydrogen. H. E. HEISSLER, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,672,528, 5.6.28. Appl., 12.3.26).—For the production of hydrogen from carbon monoxide and steam at 500–600° a catalyst is prepared by dissolving iron (150 pts.) in dilute nitric acid, adding excess of magnesite (1260 pts.) and a little potassium permanganate (25 pts.), drying the resulting paste, and converting at 500–600° into oxides, tableting, and finally heating at 800°.
C. HOLLINS.

Separation of gaseous mixtures (B.P. 292,204).—See I. **Catalysts for methyl alcohol etc.** (B.P. 293,056).—See III.

VIII.—GLASS; CERAMICS.

Colloids of clay and solutions of their sols. A. DEMOLON, H. BURGEVIN, and G. BARBIER (Compt. rend., 1928, 186, 1646–1649).—Specimens of clay which have been treated as follows: (a) saturated with calcium hydrogen carbonate, (b) half saturated, and (c) freed from bases by treatment with 0.05*N*-hydrochloric acid, and which have also adsorbed various small amounts of potassium and ammonium ions, have been extracted with 0.0071*N*-calcium hydrogen carbonate solution. It is found that samples of type (a) lose more of the adsorbed ions than either (b) or (c). The amounts extracted by four successive treatments with calcium hydrogen carbonate solution are about constant, although there is a slight increase in series (b) and a slight diminution in series (a). The amounts of ions extracted are independent of the degree of dilution within fairly large limits of variation of volume. The results are in agreement with the theoretical conclusions previously deduced (B., 1927, 654).
H. BURTON.

Plasticity of clay. L. E. JENKS (J. Amer. Ceram. Soc., 1928, 11, 317–322).—Plasticity of clay is due to the formation of a continuous external phase by the action of water, which renders gelatinous the material surrounding the non-gelatinous particles and aggregates in the clay. The effect of simultaneous peptisation and flocculation is to preserve the gelatinous envelope and prevent the change of the material into something which will not take up water to become gelatinous again when moistened. Such materials as organic matter, dissolved carbon dioxide, or electrolytes in the water or clay assist in the formation of a

sufficient amount of material capable of becoming gelatinous when moistened; they are not the direct cause of plasticity. The Bancroft test is recommended for measuring the relative plasticity of clays. F. SALT.

Spalling of silica refractory material at low temperatures. A. T. GREEN and H. EDWARDS (Trans. Ceram. Soc., 1927, 26, 185—202).—The effect is noted of the changes due to the presence of tridymite and cristobalite in seven types of silica products. Data are presented on the relative low-temperature expansion and its variation with temperature; temperature diffusivity; and the results of spalling tests. A relatively high percentage of cristobalite in a silica product favours spalling, but the influence of tridymite is much less pronounced. No direct relation was found between the diffusivity and the spalling tendency. The spalling tendency of silica refractories at low temperatures is influenced greatly by the elasticity of the product.

F. SALT.

Control of silica brick making, based on load-test indications. I. *Résumé* and theoretical. II. Control of grog fractions. III. Interdependence of matrix nature and type of industrial service. IV. Control of matrix fractions. A. J. DALE (Trans. Ceram. Soc., 1927, 26, 203—209, 210—216, 217—224, 225—230).—The sequence and nature of the changes (silica conversions) taking place during the firing of lime-bonded silica bricks are explained with the aid of a diagram. The presence of unconverted quartz in a fired silica brick causes premature failure under load at high temperatures. Bricks consisting mainly of cristobalite and glass will support considerable loads up to 1700°. Faulty firing or cooling may neutralise any benefits derived from quartz conversion. Four factors which govern the degree of conversion of quartzitic rocks into cristobalite during kiln firing are considered, viz., type of rock, fineness of grains, shape of grains, and temperature. A suitable firing schedule to give maximum quartz conversion is outlined. The nature of the matrix and its relation to the physical properties of silica bricks at high temperatures are considered in detail. Three general and comprehensive kinds of industrial usage are defined, and types of matrix for these different conditions are suggested. The advantages of a tridymite matrix are pointed out, and the methods of producing it in practice are indicated.

F. SALT.

Potassium bisulphate fusions [in analysis of aluminous refractories]. W. R. KERR (J. Amer. Ceram. Soc., 1928, 11, 330).—To avoid trouble due to spattering during the fusion, the bisulphate is subjected to a preliminary fusion in a porcelain or fused silica crucible until the melt is quiescent. The "fused buttons" thus obtained are used in making the fusions. Raw materials should always be calcined before the fusion is made. Crucibles of fused silica are best suited to this work, but well-glazed porcelain gives good results for control work.

F. SALT.

Evaluation of silicon carbide and synthetic corundum. H. DANNEEL (Chem. Fabr., 1928, 164—167).—The manufacture, properties, and various grades of the carbide and of fused aluminium oxide (artificial

corundum) are described. Chemical examination in both cases is difficult, and not necessarily conclusive; reliance must be placed on the physical properties and on microscopical examination. S. I. LEVY.

PATENTS.

Manufacture of transparent fused pure silica. QUARTZ ET SILICE (B.P. 287,522, 12.3.28. Fr., 23.3.27).—As large blocks as possible of quartzites of the type specified in B.P. 190,477 (B., 1923, 454 A) are packed in sand and heated to fusion in an electrical resistor furnace. When cold, the blocks are divided up along planes containing flaws, the best pieces being used for optical purposes, and the rest shaped or drawn for other uses.

A. COUSEN.

Manufacture of glass. E. E. FISHER (B.P. 288,267 and 288,273, [A] 24.3.28, [B] 26.3.28. U.S., [A] 5.4.27, [B] 6.4.27).—See U.S.P. 1,665,693—4; B., 1928, 405.

Glass furnaces. HARTFORD-EMPIRE Co., Assees. of P. G. WILLETTTS (B.P. 269,153, 22.3.27. U.S., 6.4.26).

Apparatus for drying industrial [ceramic etc.] products. SOC. INDUS. DE BRIQUETERIE ET CÉRAMIQUE (B.P. 275,174, 7.6.27. Fr., 30.6.26).

IX.—BUILDING MATERIALS.

Physical properties of limestones used for building in the United States. D. W. KESSLER and W. H. SLIGH (U.S. Bur. Standards Tech. Paper No. 349, 1928, 21, 497—590).

PATENTS.

Burning of cement. A. ANDREAS (B.P. 268,767, 25.3.27. Ger., 31.3.27. Addn. to B.P. 263,166; B., 1928, 368).—Hot air is blown at a relatively high pressure into the clinkering zone of a cement-burning kiln, and air cooled by spraying with water is introduced below the clinkering zone, near which the kiln consists of a metal casing with thin fire-resisting walls. The material supplied to the kiln is prepared by a wet process and is dried by the waste gases from a rotary kiln. The charge is supplied either in a non-briquetted form or in small lumps, fuel being added in the mixing or grinding mill.

W. G. CAREY.

Rotary [cement] kiln and cooler. P. T. LINDHARD, Assr. to F. L. SMITH & Co. (U.S.P. 1,675,416, 3.7.28. Appl., 12.2.26).—A series of cooling drums are secured to one end of the kiln, round its circumference. Each drum communicates through a tubular neck with one of a series of circumferential holes in the kiln, so that the clinker can be discharged by gravity into the lowermost drum. A removable bushing, which extends inwardly through the kiln lining and outwardly into the tubular neck of the drum, protects the parts from wear during discharge of the clinker.

F. G. CLARKE.

Manufacture of road materials. J. HINES (B.P. 292,871, 20.1.28).—Tar macadam is strengthened by the substitution of 20% or more of bitumen in place of tar in the tar coating, and premature hardening is prevented by spraying the material on leaving the mixer with a 50% colloidal emulsion of bitumen in water.

W. G. CAREY.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Volumetric determination of vanadium in chrome-vanadium-tungsten steels. H. H. WILLARD and P. YOUNG (Ind. Eng. Chem., 1928, 20, 764—768).—Potassium bromate has been found to oxidise vanadyl salts quantitatively in presence of ammonium salts and a definite concentration of hydrochloric acid without affecting chromic salts. The excess of bromate is destroyed by boiling and the vanadic acid titrated with ferrous sulphate. The proportion of hydrochloric acid used depends on the iron and chromium content of the sample; with 4–5 g. of chrome-vanadium steel 6 c.c. of acid (d 1.18) are used. Tungsten does not interfere with electrometric titration if kept in solution as sodium tungstate. An alternative method consists in the use of diphenylbenzidine as internal indicator. In this case after removal of bromate orthophosphoric acid and sufficient sodium acetate to react with the excess of sulphuric and hydrochloric acids present are added, followed by the indicator. A blank test to give the indicator correction is necessary. Tungstic acid if present must be completely removed and the small amount of vanadium carried down with it determined colorimetrically with phosphoric acid. A number of results are given. C. IRWIN.

Persulphate method for [determining] chromium plus vanadium in chrome-vanadium-tungsten steels. H. H. WILLARD and P. YOUNG (Ind. Eng. Chem., 1928, 20, 769–770).—It has been found (cf. preceding abstract) that sodium tungstate may be retained in acid solution if ferric salts are present, and in this form it does not interfere with the persulphate or permanganate oxidation methods for determining chromium plus vanadium. The sample is dissolved in sulphuric acid, oxidised with nitric acid, and the precipitated tungstic acid redissolved in sodium hydroxide and ferric alum solution. Electrometric titration is best, as for the use of diphenylbenzidine tungsten must be quantitatively removed. The indicator method is very suitable if tungsten is absent and is performed in a manner analogous to that described above for vanadium with bromate oxidation. C. IRWIN.

Determination of copper in iron and steel. E. ZUNDEL (Chem.-Ztg., 1928, 52, 537–538).—The material is dissolved in hydrochloric acid, and the copper precipitated from the boiling solution by means of sodium thiosulphate. The precipitate, after boiling till the liquor is clear, is collected, ignited, and weighed as cupric oxide. S. I. LEVY.

Corrosion and crystallite orientation in rolled [copper] sheets. R. GLAUNER and R. GLOCKER (Z. Metallk., 1928, 20, 244–247).—The rate of dissolution in 0.27*N*-hydrochloric acid of annealed copper sheet with a regular crystallite orientation is greater than that of similar sheet with an irregular orientation, but in 0.27*N*-acetic acid the converse is true; in the first case the time-loss in weight graphs are straight lines and in the second case smooth curves convex to the time axis. Similar irregular results were obtained with 18 other corroding solutions. Frequently the surface of the irregularly oriented sheets became roughened very

rapidly, whereas that of the regularly oriented sheets remained smooth and bright for some days, although, in both cases, dissolution proceeded regularly.

A. R. POWELL.

Production of chromium surfaces for retarding the corrosion of nickel at high temperatures. G. F. SAGER (Diss., Rensselaer Polytech. Inst., 1927, [20], 31 pp.).—For chromium-plating nickel articles the surface is first cleaned by cathodic electrolysis in a bath containing 50 g. of sodium carbonate, 15 g. of sodium hydroxide, and 5 g. of potassium cyanide per litre, using a current density of 20 amp./dm.² for 15 min. Chromium is then deposited in a bath containing 250 g. of chromium trioxide, 3 g. of chromic sulphate, and 7 g. of chromic carbonate per litre using a current density of 340 amp./dm.² The initial voltage of 5.8–6.2 drops to 3.3–3.7 volts within a few hours; the current efficiency for continuous work is about 10% and the rate of increase of thickness of the deposit 0.018 mm./hr. With a coating of half this thickness the metal remained unaffected for 30 min. in sulphur dioxide at 800°. Attempts to deposit a nickel-chromium alloy from sulphate and chloride baths yielded coatings which contained no chromium. Prolonged annealing of chromium-plated nickel wires in hydrogen and *in vacuo* at 1100–1300° results in only slight interpenetration of the two metals and the resistance to corrosion by sulphur dioxide at a high temperature is somewhat reduced. "Chromising" nickel in mixture of alumina and powdered chromium at 1050–1150° produces a highly resistant alloy layer which is unaffected by subsequent cold work. A. R. POWELL.

Age-hardening silver[–copper] alloys. W. FRAENKEL and P. SCHALLER (Z. Metallk., 1928, 20, 237–243; cf. Norbury, B., 1928, 267).—Copper-silver alloys with 3–20% Cu after quenching from 725° harden on ageing above 200° owing to the separation of sub-microscopic particles of copper from the super-saturated solid solution. The optimum ageing conditions for the alloy with 8% Cu are 2½ hrs. at 290–300°, whereby a maximum hardness of 103 is obtained with a tensile strength of 41.3 kg./mm.² The latter continues to rise very slowly with prolongation of the ageing up to 6 hrs., then slowly falls. The yield point reaches its maximum in 4 hrs. at 290°, during which it rises from 10.69 to 24.23 kg./mm.², then falls to 20 kg./mm.² in 90 hrs. The electrical conductivity decreases during about 17 hrs. at 290°, showing that precipitation of the particles of copper is still taking place, but the hardness falls during this period owing to agglomeration. No hardening effect takes place at 500° but, after quenching from 500°, some slight hardening ensues at 300°.

A. R. POWELL.

Age-hardening of silver-copper-cadmium alloys. W. FRAENKEL and L. NOWACK (Z. Metallk., 1928, 20, 243).—With up to 20% Cd silver does not harden on quenching from 700–800° and annealing at 280°. The addition of cadmium to silver-copper alloys retards considerably the age-hardening at 280° and reduces the maximum hardness obtainable; thus, for the alloy with 8% Cu and 12% Cd, maximum hardness is obtained only after 10 hrs. at 280° (cf. preceding abstract).

A. R. POWELL.

Applications of the "zinc" test for cassiterite. G. H. STANLEY (J. Chem. Met. Soc. S. Afr., 1928, 28, 249—251).—To identify cassiterite in a polished rock section the surface is covered with a paste of zinc dust and water, then with a piece of thick soft filter paper and the whole inverted into a dish containing 1:1 hydrochloric acid. In 15 min. the particles of cassiterite become coated with a film of tin. To detect cassiterite in a black sand concentrate the sand is placed on a zinc sheet and covered with hydrochloric acid; in a short time the cassiterite becomes covered with a grey film of tin and can thus be readily distinguished from other heavy dark-coloured minerals.

A. R. POWELL.

Hardness of electro-deposited nickel. D. J. MACNAUGHTAN and A. W. HOTHERSALL (Trans. Faraday Soc., 1928, 24, 387—400).—Deposits obtained from solutions containing various concentrations of nickel sulphate with ammonium sulphate, boric acid, potassium chloride, sodium fluoride, and sodium sulphate were examined. Solutions containing ammonium sulphate give rise to much harder deposits than those containing boric acid (cf. B., 1924, 519). This increase of hardness appears to be independent of nickel sulphate concentration and to be unaffected by the presence of potassium chloride. An increase of hardness in deposits from solutions containing boric acid is produced by the addition of large amounts of sodium sulphate, or the substitution for potassium chloride of an equivalent amount of sodium fluoride (cf. Blum, B., 1921, 351; 1925, 810). In neither case, however, is the hardness of these deposits as high as of those obtained in solutions containing ammonium sulphate, and certain disadvantages attend the use of baths containing sodium sulphate or fluoride. Raising the temperature of solutions from 20° to 35° results in a reduction of hardness, which is relatively less than the reduction in stress. Details are given for the preparation of very pure nickel sulphate and nickel electrodes.

L. F. GILBERT.

Viscosity [of metals] at high temperatures. J. COURNOT and M. S. SILVA (Rev. Mét., 1928, 25, 82—86; cf. B., 1926, 161; 1927, 112).—The limiting creep stress was determined for annealed wires 1 and 2 mm. in diameter of commercially pure nickel, aluminium, duralumin, and alpac. The rate of extension at various temperatures was plotted against the applied stress and the creep stress obtained by extrapolating the curves to zero rate of extension. The time required to attain a constant rate of extension varies with different materials and is affected both by temperature and by applied stress. It is much shorter for the materials tested than for copper and brass. The ratio of creep stress to ultimate stress is higher for nickel than for the other materials, and is particularly low for aluminium. Between 500° and 700° the creep stress of nickel is about twice that of mild steel. Increasing the diameter of the test specimen gives higher values for the creep stress.

C. J. SMITHELLS.

Platinum-platinum rhodium thermocouples. A. SOURDILON and ROLET (Rev. Mét., 1928, 25, 90—97).—The deterioration of platinum-platinum rhodium thermocouples, protected by metal tubes, when used in steel-treating furnaces is shown to be due to

corrosion by sulphur compounds in the furnace atmosphere. Satisfactory protection can be obtained by the use of fused silica sheaths.

C. J. SMITHELLS.

PATENTS.

[Treatment of iron for the manufacture of] apparatus for carrying out chemical and other processes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 293,077, 3.3.27).—Iron for the manufacture of chemical apparatus is heated above 900° in an atmosphere of hydrogen, carbon monoxide, or mixtures of one or both of these with water vapour and carbon dioxide to remove oxygen, sulphur, and carbon, and then coated with chromium, tungsten, aluminium, or other resistant metal.

A. R. POWELL.

Alloy steel for use as a building material. VEREIN. STAHLWERKE A.-G., Assees. of W. HÜLSBRUCH (B.P. 271,470, 17.5.27. Ger., 22.5.26).—The steel contains up to 0.35% C, 0.5—1.5% Si, up to 1.2% Mn, and 0.1—0.5% Cu. The resistance to corrosion is much greater than that of plain silicon steels.

A. R. POWELL.

Composition. [Copper-nickel-zinc alloy.] C. S. CUSSEN (U.S.P. 1,674,640, 26.6.28. Appl., 21.4.24).—Nickel, copper, and bronze are melted together under charcoal and the mixture is agitated while zinc is added. After cooling slightly, arsenic trioxide is vigorously stirred in and the alloy cast.

A. R. POWELL.

Manufacture of zinc. J. ALLINGHAM (U.S.P. 1,674,030, 19.6.28. Appl., 30.4.23).—The ore is roasted, pulped, and treated with sulphur dioxide, whereby the zinc is dissolved out as zinc sulphate and separated from the solution. The zinc sulphate is then reduced to zinc and a compound of sulphur, and the latter is treated so as to form sulphur dioxide, which is returned to the process.

M. E. NOTTAGE.

Treatment of articles being galvanised. T. A. DISSEL, Assr. to CAMERON APPLIANCE CO. (U.S.P. 1,675,926, 3.7.28. Appl., 23.12.25).—A flux is heated and the vapour passed through a mass of hot zinc-coated articles.

H. ROYAL-DAWSON.

Method of zinc-coating ferrous metal. J. G. FITZGERALD, Assr. to CENTRAL ALLOY STEEL CORP. (U.S.P. 1,675,646, 3.7.28. Appl., 30.3.25).—The metal to be coated is either heated to 456—510° and then coated in a molten spelter bath to produce a tight coat, or it is coated in a molten spelter bath and then cooled, the temperature of cooling being controlled to produce the desired character and appearance of the coating.

M. E. NOTTAGE.

Manufacture of metal-coated sheet. H. A. ROEMER and D. A. WILLIAMS (U.S.P. 1,675,134, 26.6.28. Appl., 4.10.24).—Iron or steel sheets are coated with spelter and then subjected to cold-rolling. The coating will withstand severe deformation and can be spot-welded or subjected to high temperatures without scaling or peeling.

M. E. NOTTAGE.

Electrolytic extraction of aluminium. ALUMINIUM-IND. A.-G. (B.P. 280,881, 25.10.27. Ger., 22.11.26).—In the process in which the crude aluminium or aluminium alloy forms the solid anode and the bath consists of halides of aluminium and alkali or alkaline-earth

metals having a lower m.p. than the electrodes, the density of the deposition on the solid cathode may be ensured either by the addition of metallic oxides or salts to the bath or, alternatively, when metallic chlorides are to be added, by alloying the corresponding metals to the anode or by using them in the form of separate auxiliary anodes. M. E. NOTTAGE.

Production of nickel alloys. N. V. HYBINETTE (U.S.P. 1,674,438, 19.6.28. Appl., 23.9.22).—Molten nickel is subjected to an oxidising treatment to remove impurities, then deoxidised with a substance forming a non-volatile oxide, and finally treated with the requisite quantity of alloying element. A. R. POWELL.

Recovering tin from tin-plate scraps. L. U. LA CORSA (B.P. 284,691, 23.12.27. Italy, 5.2.27).—The scrap is treated with ferric chloride whereby a solution containing stannous chloride and ferrous chloride is obtained to which ammonium chloride is subsequently added, or a mixture of ferric chloride and ammonium chloride may be used for the initial attack of the tin. The tin is recovered from this solution by electrolysis. The ferrous salt may be reconverted into the ferric salt by treatment with chlorine. M. E. NOTTAGE.

Detinning processes. Q. MARINO (B.P. 282,779, 23.12.27. Fr., 27.12.26).—Tin scrap is treated with a solution of hydrochloric acid containing oxidising salts such as chromates, dichromates, nitrates, or chlorates of potassium or sodium, or ammonium chlorate, at normal temperatures, under which conditions the base metal is not attacked. When the solution is saturated with tin, the latter is recovered either by precipitation with zinc or aluminium or by electrolysis. M. E. NOTTAGE.

Recovery of precious metals from ores. S. B. McCLUSKEY (B.P. 278,742, 7.10.27. U.S., 8.10.26).—Gold or silver ores containing iron and manganese compounds in such a form that they consume cyanide are ground in a solution of cyanide, the pulp is acidified with sulphur dioxide in a closed gas-tight circuit, and a current of air blown through to expel all the cyanogen as hydrogen cyanide and to oxidise the iron and manganese sulphides to sulphates, calcium carbonate and hydroxide are added to precipitate ferric and manganic hydroxides, and the hydrogen cyanide previously expelled is reintroduced into the pulp. A. R. POWELL.

Treatment of low-grade ores. R. ANTHOINE (B.P. 292,832, 7.11.27).—The mineral, *e.g.*, cassiterite, is treated with a reducing gas at 300–900° so as to obtain at least a superficial reduction of its particles. It is then concentrated by flotation. M. E. NOTTAGE.

Treatment of metalliferous sulphides. H. E. WETHERBEE (U.S.P. 1,674,491, 19.6.28. Appl., 14.12.26).—The sulphides are heated first under conditions such that the exothermic action is controlled, then at a higher temperature (about 538°) for the maximum sulphating action, and afterwards at temperatures below 649° until the persisting iron sulphates are decomposed, the temperature changes at each stage being relatively abrupt. M. E. NOTTAGE.

[Fuel for] ore smelting. F. HOFMANN (B.P. 293,109, 29.3.27).—Pit coal is briquetted, *e.g.*, at 150–

375°, with or without the addition of small quantities of binding material, and then semi-coked (500–600°). By modifying the working conditions of briquetting or semi-coking, a material of any desired combustibility may be obtained, which is used for smelting.

M. E. NOTTAGE.

Melting furnace. C. E. FRAZIER (U.S.P. 1,675,542, 3.7.28. Appl., 4.8.26).—A furnace of twin construction consists of melting and working chambers arranged side by side, the material under treatment being carried in its lower, and a heat-giving gaseous stream in its upper part. A port is provided at the downstream end of the melting chamber and through this a portion of the gaseous stream flowing through the furnace may be diverted. M. E. NOTTAGE.

[Electrically-heated apparatus for] case-hardening or malleabilising iron or steel articles. MORRIS MOTORS (1926), LTD., J. A. RICHARDSON, and R. A. SMART (B.P. 293,531, 21.5.27).

[Washer] apparatus for concentrating minerals. F. L. WILDER, E. SCHIFF, and E. S. KING (B.P. 293,411, 6.4.27).

Carbonising metallic charges (U.S.P. 1,674,982).—See XI.

XI.—ELECTROTECHNICS.

Chromium surfaces. SAGER.—See X.

PATENTS.

Electric furnace. M. UNGER, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,675,237, 26.6.28. Appl., 9.12.26).—A primary winding is inductively coupled with an annular channel depending from a crucible forming a ring-shaped reservoir and communicating with the inner periphery of the reservoir. J. S. G. THOMAS.

Electric furnace and method of carbonising metallic charges therein. W. E. MOORE, Assr. to PITTSBURGH RES. CORP. (U.S.P. 1,674,982, 26.6.28. Appl., 11.5.22).—Iron or other metal mixed with carbonaceous materials is melted and agitated in an arc-type electric furnace. J. S. G. THOMAS.

Shaft-type electric furnace. T. F. BAILY (U.S.P. 1,675,744, 3.7.28. Appl., 19.7.27).—Means are provided for melting a charge and feeding the molten material downward through a carbonaceous resistor extending between end electrodes within the furnace. J. S. G. THOMAS.

Production of magnetic materials. STANDARD TELEPHONES AND CABLES, LTD., Assees. of J. W. ANDREWS and R. GILLES (B.P. 269,511, 23.3.27. U.S., 17.4.26).—Finely divided particles of a magnetic alloy containing more than 25% of nickel and the remainder principally iron are coated with an insulating material, *e.g.*, a mixture containing 1½ parts of silicic acid and 1 part of water-glass, and enveloped in an insulating medium composed of 1 part of silicic acid and 1½ parts of ferric oxide, then heated, formed into a homogeneous solid, and heated, *e.g.*, at 480–950°. J. S. G. THOMAS.

Deposition of thorium [on filaments] from its vaporisable compounds. J. W. MARDEN, T. P. THOMAS, and J. E. CONLEY, Assrs. to WESTINGHOUSE LAMP Co. (U.S.P. 1,675,120, 26.6.28. Appl., 9.12.22).—

A filament of tungsten free from thorium compounds is coated thermally with metallic thorium unmixed with other metals. Apparatus is described.

F. G. CLARKE.

Electron-emitting cathodes. J. R. SINCLAIR (B.P. 289,509, 24.1.27).—An electrically conducting filament is coated with a mixture of the peroxides of barium, strontium, and calcium in the proportions 2:1:1 by wt., by immersing the filament in a bath of the materials in a state of suspension or in the colloidal state and passing a current from the filament to the containing vessel, so that deposition on the filament occurs due to electrolysis and cataphoresis. The coating is subsequently activated by being heated at about 900° in *vacuo*.

J. S. G. THOMAS.

Manufacture of [oxide] cathodes for discharge devices. ALLGEM. ELEKTRICITÄTS-GES. (B.P. 268,815, 1.4.27. Ger., 1.4.26).—A paste composed of fine metallic powder, *e.g.*, molybdenum, and a viscous non-aqueous liquid, *e.g.*, sugar dissolved in ethylene glycol, is applied to an oxide base and heated to the sintering temperature of the metal. When an easily oxidisable metal such as nickel or iron is used, the heating is carried out in an atmosphere of hydrogen or other inert gas.

J. S. G. THOMAS.

[Depolariser for dry] battery cell. H. CSANYI (U.S.P. 1,676,007, 3.7.28. Appl., 26.5.26).—Manganese hydroxide is mixed with the chloride of a tervalent metal of a higher potential than the negative electrode of the cell.

J. S. G. THOMAS.

Insulating paper (B.P. 262,828).—See V. **Aluminium** (B.P. 280,881).—See X. **Production of rubber** (B.P. 269,504 and 279,474). **Electrodeposition of rubber** (B.P. 293,095).—See XIV.

XII.—FATS; OILS; WAXES.

Influence of [area of] active surface of a nickel catalyst on the velocity of hydrogenation of oils. S. JÓZSA (Z. angew. Chem., 1928, 41, 767—771).—The velocity of hydrogenation of neutral sunflower oil increases with concentration of catalyst for all the concentrations used (1—10%). There is no apparent relationship between the reaction rate and the surface area of the catalyst, determined by Mitscheilich's method, due to the fact that as the catalysts were prepared in different ways, it was improbable that for catalysts with equal nickel content, the amount of active nickel present was the same. In order to overcome this difficulty, a single catalyst was used, but its surface was varied by using it in conjunction with different carriers. A definite quantity of nickel formate was added to the oil, and 2% of active charcoal. Experiments were carried out to show that the charcoal acted only as a carrier, the catalyst being distributed over its surface. The carriers used comprised active charcoal, two varieties of animal charcoal, blood charcoal, infusorial earth, wood charcoal, and pumice. It was found that the velocity of reaction was proportional to the surface area of the catalyst, so that the value of a catalyst can be found by determining its surface area.

A. J. MEE.

Fluorescence of Italian olive oils in Wood's light. R. STRATTA and A. MANGINI (Giorn. Chim. Ind. Appl., 1928, 10, 205—207; cf. Baud and Courtois, B., 1928,

201).—When exposed to Wood's light in layers at least 3 mm. thick, pressed virgin Apulian oils acquire a lemon-yellow to orange fluorescence changing, after an exposure of 30 min., to natural sienna-earth colour. Under similar treatment, Sansa oils, extracted by solvents and refined, give a sky-blue fluorescence, unchanged by protracted exposure to the light, whilst refined lampante (washed and filtered) oils assume a dull, greyish-blue fluorescence. The presence of 1% or more of refined oil in virgin oil changes the colour of the fluorescence, which becomes almost white with 10%, pale blue with 30%, and brilliant sky-blue with 60—100% of the refined oil. A characteristic red band, with its mid-point at 669 μ , is always present in the fluorescence spectrum of crude olive oil, and serves for the recognition of as little as 10% of virgin oil in refined oil. T. H. POPE.

Analysis of edible oils. A. FALCONE (Annali Chim. Appl., 1928, 18, 273—286).—Last season furnished a poor yield of olive oil of low quality, mainly owing to the prevalence of *Mosca olearia*, the larvæ of which eat the fruit and kill it before maturation. For the detection of adulteration of the oil with seed-oils, use is made of the density, the thermo-sulphuric constant determined by means of the Tortelli thermo-oleometer, and the refractometer reading (Zeiss butyro-refractometer at 25°). The following are the maximum values obtained with 50:50 mixtures of olive oil and various seed-oils for the density, thermo-sulphuric constant, and refractometer reading respectively: Arachis 0.9170, 49, 64; sesamé 0.9191, 54, 65; colza 0.9158, 51, 65.8; ravisson 0.9157, 51.7, 66; soya 0.9202, 70, 68; cottonseed 0.9199, 59, 65.5; maize 0.9189, 59, 66.2; grape-seed 0.9200, 64, 67.5; poppyseed 0.9210, 68.5, 68.

T. H. POPE.

Washing sulphonated oils with salt solutions. C. RIESS (Collegium, 1928, 298—301).—Salt solutions, after use in washing sulphonated oils, contain more free acid than corresponds with the sulphuric acid determined gravimetrically, since interaction of the salt with the sulphonated oil yields sodium soaps and hydrochloric acid. Experiments show that for sulphonated oleic acid and saturated solutions of common salt this reaction is quantitative. The aqueous solutions of sulphonated oils which have been washed several times with sodium chloride solution are neutral to methyl-orange (about p_H 4.0). The aqueous solution of the sulphonated oil can be titrated with methyl-orange as indicator. The degree of neutralisation can be ascertained from the p_H value of the aqueous solution.

D. WOODROFFE.

Determination of ammonia in sulphonated oils. G. LANG (Ind. Eng. Chem., 1928, 20, 693).—To an emulsion of 10—15 g. of sulphonated oil with 50 c.c. of water are added, with continuous shaking, 100 c.c. of 4% sodium hydroxide solution followed by 100 c.c. of 8% calcium chloride solution. A gummy calcium soap is precipitated and the ammonia can be rapidly distilled off into standard acid without bumping or frothing. The results obtained by this method are in agreement with the more tedious earlier methods. S. S. WOOLF.

Fatty acid compounds in leather. SCHINDLER and KLANFER.—See XV. **Cacao butter.** ALBERS.—See XIX.

PATENTS.

Extraction of oils, fats, etc. from [waste] animal or fish matter. J. LEWIS (B.P. 292,327, 22.12.26).—The material is subjected to a preliminary heating during maceration, further heated in an intermediate chamber at well above 100°, and then jets of steam or hot air play upon it while subject to the action of a centrifugal separator. E. HOLMES.

Rendering of animal fatty tissues. E. C. R. MARKS. From PERFECTION Co., INC. (B.P. 292,719, 4.4.27).—An apparatus is described whereby a thin layer of waste fatty material, in the finely-divided and plastic condition, may be treated by moderate heat and mechanical pressure to give an edible fat. E. HOLMES.

Apparatus for extracting fats at low temperatures. A. PANSKY (B.P. 293,176, 2.6.27).—A low-pressure apparatus has been devised whereby oleaginous seed, hides, tannery waste, bones, or wool may be continuously extracted with the vapours of volatile solvents at comparatively low temperatures. The material is contained in a digester and rests on a false bottom above a steam chest for vaporising the solvent. The solvent passes through a condenser to a still the vapours from which are returned to the digester. E. HOLMES.

Treating oils, fats, emulsions, etc. T. D. KELLY (B.P. 293,255, 28.11.27).—It is claimed that the treatment of oils, fats, and emulsions containing upwards of 5% of protein or amyloid products with ultra-violet or other radioactive rays simultaneously with, or subsequent to, treatment with β -rays, will induce reactions to produce vitamins or enzymes in substances intended for food or for external use as an ointment. E. HOLMES.

Soap stabilisation. J. R. POWELL, Assr. to ARMOUR & Co. (U.S.P. 1,672,657, 5.6.28. Appl., 24.12.26).—The condensation product of an aromatic amine (aniline) with an aldehyde (acetaldehyde) is mixed with soap at any desired stage of manufacture in order to decrease the tendency of the soap to become rancid. The amount used is 0.02–0.5%. C. HOLLINS.

Detergent, cleansing, and polishing compositions. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, and E. CHAPMAN (B.P. 286,197, 21.9.26).—Mixtures of sulphonic acids of aromatic hydrocarbons (cf. B.P. 274,611 and 279,990; B., 1927, 841; 1928, 50) or their salts in the form of aqueous solutions or of pastes with organic solvents, *e.g.*, benzene, naphtha, light petroleum, chlorinated hydrocarbons, etc., and, if desired, thickening, tinting, or abrasive materials, soap, or sulphonated oil, are claimed. S. S. WOOLF.

Detergent compositions. HENKEL & Co., G.M.B.H. (B.P. 276,339, 16.8.27. Ger., 17.8.26).—A "self-acting" detergent for delicate and coloured textiles consists of carbamide, a carbamide-decomposing enzyme (*e.g.*, urease), and tryptic or pancreatic enzymes, as well as known soaking and alkaline agents. S. S. WOOLF.

Fats from wool (B.P. 275,627).—See V.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Effect of weathering on the heat-reflecting efficiencies of paints on metal tanks. H. A. NELSON and R. W. JAMIESON (Ind. Eng. Chem., 1928, 20, 702—

705).—Comparative tests extending over 11 months were carried out with small tanks filled with petrol, fitted with thermometers and breathing tubes, and exposed to the weather, the variation between the records of an exposed thermometer near by and the temperatures within the tanks being taken as the measure of the efficiency of the paint. The change in efficiency of black paint was negligible but zinc paint, aluminium paint, and non-chalking white enamel showed losses of 50–60% efficiency. A gradually chalking white paint showed little loss. Evaporation losses with these weathered paints were almost identical with the exception of the white paint above mentioned, in which case they were less than half that of the others. C. IRWIN.

Gold paint manufacture. F. CHEMNITZ (J. pr. Chem., 1928, [ii], 119, 215–217).—A two-operation method of manufacture from ready-made ingredients is described. C. HOLLINS.

Accelerated tests for the settling of pigments in paints. S. WERTHAN and R. H. WIEN (Ind. Eng. Chem., 1928, 20, 729–732).—From consideration of the factors influencing the settling out of ready-mixed paints an accelerated test is developed consisting of a standard cycle of heating at 65–70° and centrifuging at 1000 r.p.m. The degree of settling is noted, and the consistency and nature of the sediment are examined by penetrometer readings and determinations of the ease of reincorporation. The results of such accelerated tests on flat wall paints agree in the main with "period" settling tests. S. S. WOOLF.

Settling of pigments in house paints. H. I. BEAKES (Ind. Eng. Chem., 1928, 20, 732–734).—A preliminary report on a series of tests on single- and mixed-pigment paints, from which, by using containers fitted with suitable apertures, samples may be drawn at any time from different levels, and their pigment content determined. The thinning caused by mixing certain single-pigment paints at the same consistency is demonstrated. S. S. WOOLF.

Laboratory apparatus for preparing duplicate uniform paint, varnish, and lacquer films. J. C. BRIER and A. M. WAGNER (Ind. Eng. Chem., 1928, 20, 759–764).—The device comprising a doctor blade, which can be adjusted for height by means of a micrometer screw, ensures uniform thickness, and a basket attachment completely encloses the fluid sample, thus preventing more than a negligible loss of volatile matter. W. J. POWELL.

Application of methods of dyestuff analysis to the examination of pigments and lakes. A. G. GREEN (J. Oil Colour Chem. Assoc., 1928, 11, 38–48).—A discussion and description of a scheme for the identification of organic dyestuffs in pigments and lakes. The scheme is based on principles already described. Unsulphonated dyestuffs of the nitro- and azo class, vat dyes of the indigoid class and many of the anthraquinone class are removed by extraction with boiling benzene and differentiated in known ways. Colour insoluble in hot benzene is extracted successively with 2% sodium carbonate, which removes acid, acid mordant, or salt (direct) dyestuffs, and sodium hydroxide in saturated salt solution, which

decomposes the lakes of basic dyestuffs and removes the lake-forming acid. If the lake is undecomposed or does not give a coloured extract with formic acid or dye wool after this treatment with sodium hydroxide, a fresh sample is boiled with 3% hydrochloric acid, when the presence of a mordant dye is indicated by change of colour. Vat dyes of the anthraquinone series, sparingly soluble in water, may be detected by the dyeing of cotton when boiled with sodium hyposulphite and dilute sodium hydroxide. Differentiation of dyestuffs within these groups is effected in known ways. Azo dyes of the pyrazolone series are characterised by the colourless solutions obtained on reduction, which become violet when oxidised by air. Azo dyes containing only a carboxylic and no sulphonc group are usually soluble in acetone. R. BRIGHTMAN.

Theories of pigment settling. P. R. CROLL (Ind. Eng. Chem., 1928, 20, 734—735).—The influence on pigment settling of particle size and shape, viscosity and yield value of the vehicle, and degree of wetting of pigment by vehicle is briefly indicated. S. S. WOOLF.

Characteristics and treatment of insulating varnishes. P. B. COCHRAN and H. J. GRAHAM (Ind. Eng. Chem., 1928, 20, 698—701).—A saving in drying time of varnish may be effected by the use of ozonised air and elevated temperatures in the baking process, and an initial baking of the unvarnished piece in ozonised air also greatly accelerates the process. Treatment of cotton material with ozone under suitable conditions causes an increase in tensile strength of 20—30%, and such a process may be economically feasible where increased tensile strength is desirable. Several different methods for determining hardness of varnish films have been examined, the most accurate and convenient being the swinging-beam hardness tester. W. J. POWELL.

Formulation of nitrocellulose lacquers. H. E. HOFMANN and E. W. REID (Ind. Eng. Chem., 1928, 20, 687—693).—A study of the effect of changes in composition of pigmented nitrocellulose lacquers on the physical properties of the finished surface. The results are recorded diagrammatically by means of triangular co-ordinate charts which may be used to formulate new lacquers suitable for particular purposes. W. J. POWELL.

Characterisation of Peru and Tolu balsams, and of storax. L. ROSENTHALER (Pharm. Ztg., 1928, 73, 837—839).—The iodine values of the alcohols present in the ether-soluble components of Peru and Tolu balsams, and extracted from these by hydrolysis with sodium hydroxide, have been determined for a large number of samples. In the case of storax, the soluble components are best extracted with light petroleum, the hydrolysis and determination of the iodine value being then carried out as before. S. I. LEVY.

PATENTS.

Manufacture and application of water paints and the like. BRIT. DYESTUFFS CORP., LTD., A. SHEPHERDSON, and E. CHAPMAN (B.P. 285,929, 26.8.26).—Easily miscible and readily flowing water paints, distempers, etc. are made by the addition to the usual ingredients of 0.1—1.0% of a wetting-out or dispersing agent. S. S. WOOLF.

Conversion of insoluble substances [resins, dyes, etc.] into a state of aqueous solution or suspension. I. G. FARBENIND. A.-G. (B.P. 264,860, 20.1.27. Ger., 20.1.26).—The substance (e.g., resin, insoluble dye, lake, lacquer, etc., but excluding sulphur) is dissolved in a solvent substantially immiscible with water, and the solution is emulsified in water with the aid of aromatic sulphonc acids or sulphonates (excluding sodium sulphanilate and sulphite-cellulose pitch), especially such as are wetting-out agents, e.g., isopropyl-naphthalenesulphonc acid, sodium dibutylsulphanilate, sulphostearic anilide, etc. Glue or other protective colloid may be added to facilitate dispersion. The emulsions of dyes or lakes may be used for dyeing all classes of material. C. HOLLINS.

Manufacture of phenol-formaldehyde condensation products. G. BIA and J. E. D. DE GRANVILLE DE BIELIZE (B.P. 292,629, 17.1.27).—A mixture of phenol or cresol with a quantity of formaldehyde greatly in excess of the equimolecular proportion is boiled for 1 hr. or less in the presence of a catalyst, e.g., magnesia, or sodium carbonate or borate, after which it is cooled to 50°, the catalyst is neutralised if alkaline, and the resinous condensation product is removed, heated at a temperature above 100°, and finally moulded at 90—112°. The liquid resin may be kept for a long time or treated with a weak electric current before the final heating and moulding. Paraffins, casein, dyes, absorbent material, etc. may also be added to the initial mixture. L. A. COLES.

Resinous condensation products. BRIT. THOMSON-HOUSTON CO., LTD., H. W. H. WARREN, R. NEWBOUND, and A. T. WARD (B.P. 292,646, 25.2.27).—Fusible resins prepared from polyhydric alcohols, e.g., glycerol, and aromatic polybasic acids, e.g., phthalic acid, are heated at 220—300° until an infusible product resembling rubber or leather is obtained, then at 170—200°, and are finally moulded at 120—170°. L. A. COLES.

Resinous reaction product of carbamide and formaldehyde. F. LAUTER, ASST. to ROHM & HAAS Co. (U.S.P. 1,672,848, 5.6.28. Appl., 12.1.26).—*p*-Toluenesulphonamide is added to carbamide (etc.) for condensation with formaldehyde to give a resin having less tendency to crack, and greater resistance to heat. C. HOLLINS.

Manufacture of condensation products from urea, thiourea, or their derivatives and an alcohol or ketone. I. G. FARBENIND. A.-G. (B.P. 278,390, 4.10.27. Ger., 4.10.26).—Oily or resinous condensation products are obtained by heating under pressure urea, thiourea, or their derivatives with excess of an alcohol or ketone (benzyl alcohol, diacetin, acetophenone, etc.) in the presence, if required, of a catalyst, e.g., glycerol, strong mineral acids, etc. S. S. WOOLF.

Treatment of articles and masses obtained from phenol-aldehyde condensation products. G. PETROFF (B.P. 292,636 and Addn. B.P. 262,637, 18.2.27).—(A) The surface layer of articles constructed from the condensation products is coloured and rendered resistant to attack by the atmosphere by immersing the articles in hot or cold solutions of dyes and/or protective agents (e.g., quinol, aminophenols, tannin, resins, shellac, cellulose ethers, etc.) in alcohols, alde-

hydres, lower ketones, or volatile organic acids. The dyes etc. may be fixed, *e.g.*, by treatment with dichromate solution. (b) The articles are treated with aqueous solutions of phenols, sulphonic acids, or sulphonated oils, with or without the addition of organic solvents, then with aqueous solutions of dyes, and finally, if desired, with formaldehyde and/or other fixing agents. L. A. COLES.

Method of preparing white paint or pigment. L. WILLIAMS (B.P. 293,661, 13.2.28).—See U.S.P. 1,662,625; B., 1928, 308.

Cellulose acetate lacquers (B.P. 292,398). Resin-coated board (B.P. 291,633).—See V.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Thermochemistry of rubber. S. BOSTRÖM (Kolloidchem. Beih., 1928, 26, 439—470).—Details are given of an apparatus for studying the thermal changes of slow reactions by means of a differential calorimetric method. The relative activities of technical rubber fillers may be determined by measuring the heat of adhesion between the filler and caoutchouc. Measurements are recorded of the heat of wetting of various samples of soot by benzene, and the heat of swelling of caoutchouc and of caoutchouc-soot mixtures in benzene, and the validity of the method is tested by comparison of the thermal data with previously recorded mechanical data. The stretching and releasing of raw rubber is investigated thermodynamically, and experiments are described for measuring the change in the total energy and the temperature coefficient of the mechanical work. The discrepancy between theory and experiment is discussed. The thermal changes accompanying vulcanisation are considered, and the heat values of the reaction between sulphur monochloride and rubber dissolved in benzene are determined for a concentration ratio of 0—1.3 g. of sulphur monochloride to 1 g. of rubber. L. L. BIRCUMSHAW.

Tensile properties of soft rubber compounds at temperatures ranging from -70° to $+147^{\circ}$. R. F. TENER, S. S. KINGSBURY, and W. L. HOLT (U.S. Bureau Stand. Tech. Paper 364, 1928, 22, 367—377; cf. van Rossem, B., 1926, 502).—Tensile strength, elongation, and tensile product over a range of temperatures between -70° and 147° are recorded for six vulcanised mixtures, representing different degrees and types of compounding. At low temperatures, *e.g.*, below -60° , the rubber becomes rigid with an increased tensile strength, whereas at high temperatures it has practically no tensile strength. The differences arising from compounding are in degree rather than in kind. The tensile product between 80° and -40° was fairly constant for a mixture of rubber (100), sulphur (1.91), zinc oxide (1.03), and tetramethylthiuram disulphide and somewhat less so for a mixture of rubber (100), sulphur (3), zinc oxide (5), carbon black (40), stearic acid (3), and mercaptobenzthiazole (0.6), whereas a mixture of rubber (92.5) and sulphur (7.5) was most affected. D. F. TWISS.

Sulphur in rubber manufacture. D. F. TWISS (Trans. Inst. Rubber Ind., 1928, 3, 386—400).—The fineness, solubility, and transformations of various forms of sulphur are discussed, and their bearing on manufac-

turing processes is indicated. When heated in admixture with rubber S_{μ} rapidly changes to soluble sulphur; for this and other reasons it is improbable that the effect produced by vulcanisation is caused merely by the formation of a fine dispersion of insoluble S_{μ} in chemically unchanged rubber (cf. Dannenberg, B., 1927, 564). D. F. TWISS.

Carbon blacks and their use in rubber. II. Experiments in vulcanised rubber. N. GOODWIN and C. R. PARK (Ind. Eng. Chem., 1928, 20, 706—715; cf. B., 1928, 579).—Physical tests have been made on rubber containing various proportions of the five types of carbon black mentioned earlier and vulcanised with the aid of different accelerators, *viz.*, mercaptobenzthiazole, diphenylguanidine, hexamethylenetetramine, ethyldeneaniline, and litharge. The standard mixture was rubber 100, zinc oxide 5, sulphur $3\frac{1}{2}$, with addition of accelerator, stearic acid, and 10—40 vols. of carbon black. With mercaptobenzthiazole the mixtures containing the various blacks fall in practically the same order with respect to hysteresis, hardness, and time of best vulcanisation as do the corresponding blacks amongst themselves with respect to adsorptive power and inverse of particle size and as the unvulcanised mixings with respect to plasticity. Tensile strength and resilient energy, however, do not fall in this order, the channel black produced for use in rubber giving the highest values, whilst the still finer material produced for varnish gives the lowest; if particle size is of importance it must be masked here by some other factor. The abrasion resistance of the various vulcanised mixtures is comparable for all except for the black produced by thermal decomposition of natural gas—this is much inferior to the others; the abrasion resistance in all cases is improved by increase in the proportion of black in the rubber even beyond the point of maximum tensile strength or resilient energy. When using channel blacks, the additional presence of stearic acid is beneficial with respect to modulus and abrasion resistance of the vulcanisates, but the effect appears not to be directly due to the acid or to resulting zinc stearate. It is remarkable that the order of the results is not necessarily the same with the use of other accelerators. With ethyldeneaniline the black produced by incomplete combustion of natural gas at high temperatures gave the highest tensile strength and abrasion resistance, whilst the highest tensile product at best cure was given by the black obtained by thermal decomposition. No other accelerator gave products with as high resistance to abrasion as mercaptobenzthiazole. D. F. TWISS.

PATENTS.

Electrodeposition of rubber from latex etc. W. A. WILLIAMS (B.P. 293,095, 28.3.27).—A metallic salt, such as zinc sulphate, is added to ammoniacal latex, vulcanised or unvulcanised, without causing coagulation, and electrodeposition of the rubber is effected using both electrodes of the same metal, *e.g.*, zinc, with an external voltage not exceeding 1.5 volts. D. F. TWISS.

Preservation of rubber latex. I. G. FARBENIND. A.-G. (B.P. 271,863, 18.5.27. Ger., 28.5.26).—Organic amines soluble in water and of an alkalinity at least

equal to that of ammonia, *e.g.*, mono-, di-, or tri-alkylamines, cyclohexylamine, benzylamine, or trihydroxyethylamine, or mixtures of these with one another or with ammonia, afford much more effective protection against coagulation than does ammonia.

D. F. TWISS.

Drying products produced from aqueous dispersions of organic substances, particularly of caoutchouc or caoutchouc-like substances, by agglomeration upon a base. ANODE RUBBER CO. From P. KLEIN (B.P. 293,061, 25.1.27).—In drying deposits from aqueous dispersions of organic substances, especially of rubber, on moulds the temperature is maintained higher on the side of the deposit adjacent to the mould, while a current of dry or moist air is applied to the cooler exposed surface. Such procedure prevents skin-formation and consequent retardation of the evaporation.

D. F. TWISS.

Direct production of homogeneous rubber goods from rubber dispersions by electrical means. ANODE RUBBER CO., LTD. (B.P. 269,504 and 279,474, [A, B] 21.3.27. Hung., [A, B] 15.4.26).—(A) The porous moulds, impregnated with electrolyte, which are used for receiving the electrophoretic deposit of rubber (B.P. 223,189; B., 1925, 46) give less satisfactory service after repeated use, but can be restored to good condition by heating, by immersion in a colloid-free electrolytic bath and passing a current through the mould in the opposite direction, or by rinsing under pressure with an alkaline solution, *e.g.*, of ammonia, or with an electrolyte solution in such a way that the solution passes from the anodic side to the cathodic side. (B) In the electrophoretic deposition of rubber on a porous mould surrounding the anode, increased firmness of the deposit is ensured by adding to the electrolyte solution, which is used for impregnating the mould, substances capable of yielding agglomerating ions, *e.g.*, calcium sulphate or a mixture of a calcium and a magnesium salt. If, in the course of repeated use, calcium carbonate becomes precipitated in the pores or on the surface of the mould, immersion in an acid bath, preferably of hydrochloric acid, restores uniform porosity.

D. F. TWISS.

Manufacture of articles from aqueous dispersions of rubber and similar vegetable resins or compounds thereof. DUNLOP RUBBER CO., LTD., and G. G. THORNTON (B.P. 292,704, 29.3.27).—Rubber articles obtained in a raw condition from natural or artificial latex, compounded or otherwise, by dipping or electro-deposition are dried and then subjected to vulcanisation between engraved plates, applied with slight pressure if desired, in boiling water or a boiling aqueous solution. The articles receive an impression of the pattern from the plates.

D. F. TWISS.

Manufacture of rubber articles. S. D. SUTTON, and VEEDIP, LTD. (B.P. 293,111, 30.3.27).—In the manufacture of rubber articles by dipping suitably shaped formers into latex, whether vulcanised or unvulcanised, a film of suitable thickness is first produced. The former, coated with this dry film, is then given a further "dipped" layer of thickened or concentrated latex, which is then heated so as to produce a dry external skin covering a layer which is still moist or

liquid. In this condition the article is subjected to the action of a rubber solvent or softener, *e.g.*, naphtha, benzene, or carbon tetrachloride, which causes a roughening or wrinkling of the surface. Drying is then completed and, if desired, a final coat of thin latex applied and vulcanisation effected. Articles may thus be produced with an ornamental non-slip surface.

D. F. TWISS.

Treatment of rubber stock. H. R. MINOR, Assr. to GEN. CARBONIC CO. (U.S.P. 1,675,124, 26.6.28. Appl., 11.9.26).—Solid carbon dioxide is used to control the temperature of rubber during processing.

D. F. TWISS.

Manufacture of sponge rubber. T. W. MILLER, Assr. to FAULTLESS RUBBER CO. (U.S.P. 1,674,053, 19.6.28. Appl., 22.9.27).—A mixture of unvulcanised sponge rubber stock with particles of vulcanised sponge rubber to which additional "blowing" agent has been added is shaped and vulcanised.

D. F. TWISS.

Regeneration of rubber. W. SCHEITHAUER (B.P. 270,675, 20.4.27. Ger., 8.5.26).—Scrap rubber, with or without fibrous constituents, is regenerated by heating under pressure with a solution of sodium hydroxide which has been in intimate contact with a cellulosic substance, *e.g.*, wood pulp, sawdust, or lignocellulose, or which has issued as a waste product from a manufacturing process such as that of viscose. Such solutions enhance and accelerate the regenerating process.

D. F. TWISS.

Manufacture of coloured rubber goods. I. G. FARBERIND, A.-G. (B.P. 277,034, 5.9.27. Ger., 4.9.26).—Coloured rubber is produced by incorporating, prior to vulcanisation, an insoluble metal salt or lake derived from a triarylmethane dye containing a sulfo-group in an *ortho*-position to the methane carbon atom or from a sulphonic or carboxylic acid of an anthraquinone compound with at least one free or substituted amino-group. These are exceptions to the general instability of the triarylmethane and anthraquinone colours under the conditions of vulcanisation.

D. F. TWISS.

Production of jelutong products. M. L. HAMLIN, Assr. to BEECH-NUT PACKING CO. (U.S.P. 1,674,435, 19.6.28. Appl., 4.11.27).—Coagulated latex from *Dyera costulata* or similar trees is heated and mixed with an anti-oxidant so as to render it substantially resistant to atmospheric oxidation.

D. F. TWISS.

Vulcanisation of rubber. A. C. BURRAGE, JUN. (U.S.P. 1,677,169, 17.7.28. Appl., 8.2.24).—See Can. P. 245,929 and 245,930; B., 1926, 453.

XV.—LEATHER; GLUE.

Chromium-fatty acid compounds and their occurrence in leather. W. SCHINDLER and K. KLANFER (Collegium, 1928, 286—291).—Four compounds of chromium and fatty acids have been isolated. The α -compound was prepared by boiling chrome alum solution with solutions of sodium salts of different fatty acids. The product was sparingly soluble in trichloroethylene (violet solution) and in carbon tetrachloride, was quite resistant to hydrochloric acid, but was decomposed by hot 30% alkali. It is not a chrome soap but a complex compound similar to lead oleate, and containing 7.00—7.52% Cr. The same chromium content was found in the products obtained from fish oils and stearic and oleic

acids. β -Compounds were obtained by the action of soap solutions on chrome alum in the cold; they gradually revert to the α -type. Trichloroethylene solutions of the β -compounds are green. γ -Compounds are present in the residue left after trichloroethylene extraction of the precipitate obtained on boiling mixed solutions of soap and chrome alum. Some fatty acids are liberated from them by treatment with hydrochloric acid. A greater amount is formed at higher basicities. The presence of γ -compounds in leather is very probable. They cannot be extracted with carbon tetrachloride. δ -Compounds can be obtained by treating cold, freshly precipitated chromium hydroxide with oleic acid. They contain 15.9–16.04% Cr, are slightly soluble in trichloroethylene, and readily decomposed with boiling hydrochloric acid. Aged chromium hydroxide does not form γ -compounds, but chrome-tanned leathers yield to carbon tetrachloride an extract containing 0.38–1.71% Cr, and on extraction with acetone this yielded a residue with 4.75–7.0% Cr. D. WOODROFFE.

Cacao tannin. JENSEN.—See XIX.

PATENTS.

Emulsions suitable for tanning. RÖHM & HAAS A.-G. (B.P. 280,509, 22.9.27. Ger., 13.11.26).—An insoluble oil or fat (*e.g.*, neatsfoot oil) is intimately mixed with a freshly-precipitated metallic hydroxide (*e.g.*, aluminium hydroxide), with or without the addition of urea, and water to form an emulsion, which may be diluted with water as required. D. WOODROFFE.

Treatment of fabric to resemble wash leather. H. W. ROWSELL, W. JANVIER, and SHAMMY Co., LTD. (B.P. 289,592, 24.2.27).—A coloured absorbent fabric which is finished with a raised nap is impregnated with a solution of albumin and when dry is tanned. The tanned fabric is then washed, passed through squeezing rolls, and dried. D. J. NORMAN.

Manufacture of imitation leather or skins [by coating fabric with cellulose acetate]. C. E. SIMPSON (B.P. 291,890, 12.3. and 9.7.27).

Scraper for separating solidified colloids from supporting surfaces. C. GREINER (B.P. 292,575, 28.12.27. Ger., 24.6.27).

Fertilisers (B.P. 293,168).—See XVI.

XVI.—AGRICULTURE.

Soil-moisture conditions in relation to plant growth. F. J. VEIHMAYER and A. H. HENDRICKSON (Plant Physiol., 1927, 2, 71–82).—The movement of water from moist to drier soils was only 8 in. in 139 days. No relation was observed between soil moisture content (above the wilting point) and either the rate of use of water or the elongation.

CHEMICAL ABSTRACTS.

Formation of water-soluble arsenic in sulphur-lime dry mix spray. J. M. GINSBURG (J. Econ. Entomol., 1927, 20, 625–631).—The combined insecticide and fungicide known as sulphur-lime dry mix spray contains sulphur, calcium hydroxide, lead hydrogen arsenate, and “Kayso” (a mixture of casein and partly carbonated lime). A study of the influence of the

various constituents on the formation of water-soluble arsenic compounds from the lead arsenate indicated that calcium carbonate was chiefly responsible for this reaction. The use of an excess of calcium hydroxide and the substitution of casein for “Kayso” is recommended to reduce the risk of injury to foliage from water-soluble arsenic.

C. T. GIMINGHAM.

Preparation of a special light sodium fluosilicate and its use as a boll-weevil poison. H. W. WALKER (J. Econ. Entomol., 1928, 21, 156–164).—In the manufacture of sodium fluosilicate for use as an insecticidal dust, the comparatively high apparent specific gravity can be reduced by the incorporation of 10–20% of colloidal silica, giving a product with much increased covering power which causes no appreciable damage to plant tissues. The special “light” sodium fluosilicate is at least as toxic to the cotton boll-weevil as commercial calcium arsenate.

C. T. GIMINGHAM.

Preparation of special calcium arsenates containing less than 40% As (as pentoxide), and their use as boll-weevil poisons. H. W. WALKER (J. Econ. Entomol., 1928, 21, 165–173).—A special form of calcium arsenate for use as an insecticide dust, containing only about 20% of arsenic as As_2O_5 , is prepared by heating arsenious oxide with precipitated calcium carbonate in the presence of an excess of air at about 650° for 15–60 min. Calcium arsenates containing up to 57.6% of arsenic as As_2O_5 can be made in this manner. Conversion of the arsenic into the quinquevalent state is practically complete and scarcely any free calcium oxide is formed. On a commercial scale, any arsenic lost is recoverable. The product containing 20% As consists of particles having an inert core of calcium carbonate coated with substantially pure normal calcium arsenate. Laboratory and field tests showed it to be as effective in controlling cotton boll-weevil as ordinary commercial calcium arsenate containing about 40% As.

C. T. GIMINGHAM.

PATENTS.

Production of fertilisers from waste nitrogenous material. H. R. READ (B.P. 293,168, 21.5.27).—Nitrogenous material, *e.g.*, leather, shoddy, fish, glue, etc., is heated with dilute aqueous ammonia above 100° under raised pressure until the nitrogen has been partially or completely converted into a soluble form. The product is dried and ground to a meal.

L. A. COLES.

Manufacture and treatment of colloidal materials [fungicides, insecticides, etc.]. J. RILEY & SONS, LTD., W. H. BENTLEY, and W. M. COATES (B.P. 293,084, 23.3.27).—The products are obtained by mixing in the presence of glue, gelatin, etc. substances capable of yielding the desired colloidal material, *e.g.*, an acid and a polysulphide for the production of sulphur, or sodium arsenate and lead nitrate for the production of lead arsenate, and the salt formed simultaneously is removed partially by allowing it to effloresce from the resultant jelly, or completely by cutting the jelly into small pieces and soaking it in four or five changes of cold water. The jelly is dissolved in hot water and diluted as required.

L. A. COLES.

XVII.—SUGARS; STARCHES; GUMS.

Boiling-up of thin juice. V. MAJER (Z. Zuckerind. Czechoslov., 1928, 52, 465—479; see also B., 1927, 395).—If the carbonation of beet juices is carried beyond the correct alkalinity (0.01% CaO) so that more or less bicarbonate is formed, the precipitation of calcium carbonate which occurs on subsequent boiling is much slower and less complete than would be the case with pure solutions of calcium bicarbonate. This delayed precipitation is largely due to the influence of sugar and amino-acids. Boiling for 20—30 min. is necessary to remove most of the precipitable lime compounds. By working to an alkalinity corresponding to 0.01% CaO in the carbonated juice the total lime content of the juice and the amount precipitated on boiling for 30 min. are both reduced to a minimum. A deviation of 0.01% CaO from this optimum alkalinity, in either direction, increases considerably the amount of precipitate (chiefly calcium carbonate) produced on boiling, and therefore increases the amount of evaporator incrustation unless the juice is boiled up before entering the evaporating plant. J. H. LANE.

Lime-free sugar solutions. L. VAN DER HEIDE (Chem. Weekblad, 1928, 25, 274—277).—The effect of using sodium hydroxide, and of subsequent treatment with carbon dioxide, to remove calcium compounds from sugar solutions in course of treatment, in order to avoid separation of precipitates and deposits during evaporation, is considered under various conditions, and it is calculated that the necessary alkali can usually be added without much influence on the hydrogen-ion concentration of the evaporated solution, so that decomposition of the sugars is not thereby increased. Results of calculation for various conditions are given.

S. I. LEVY.

Inversion losses in gur sugar manufacture in North Behar. J. H. HALDANE (Int. Sugar J., 1928, 30, 363—365).—In the refining of gur, losses of 8—12% of the total sucrose were found to be mainly due to inversion during the filtration, storage, and evaporation of the gur syrups, owing to the practice of liming them only to slight acidity to litmus (p_H 5.4). By liming to p_H 7 the losses were reduced to 2.2%.

J. H. LANE.

Modified scale for Pfund colour grader for use on dark syrups. J. J. WILLAMAN (Ind. Eng. Chem., 1928, 20, 701).—Some syrups are too dark for the normal range of this instrument. When one to three glass plates were added the relation of the colour values so found to the number of plates was logarithmic. In this way three supplementary scales were prepared extending the upward range of the instrument from 14.0 to 15.7.

C. IRWIN.

Influence of humidity on the elastic properties of starch film. F. T. PEIRCE (J. Text. Inst., 1928, 19, 237—252 T).—Some sensitive, convenient, and non-destructive tests of rigidity—i.e., resistance to twisting and bending—are described, which have been applied to films of farina, but are also suitable for measuring the flexibility of yarns or fabrics and the effect thereon of sizes or finishing processes. The change in torsional rigidity with humidity is in agreement with the rectilinear relation deduced from the two-phase theory of moisture

absorption developed in the discussion of the corresponding effect in cotton hairs. The rate of change of flexural rigidity is nearly the same from 30% to 80% R.H., whilst the effect of humidity on the Young's modulus is expressed by sigmoid curves similar to that obtained for the rigidity of cotton, but with a lower rate of change. The torsional and flexural rigidities vary together over most of the range 34—65% R.H., and it is concluded that the test of resistance to twisting provides adequate evidence on the effects of moisture on the elastic properties generally. The amount of swelling is different in different directions; the thickness of the film increases at a rate 70% greater than the length, indicating that water goes between rather than into the layers parallel to the surface. The effects observed may be interpreted by postulating a structure analogous to that of oil and grease films, namely, that the large unsymmetrical molecules tend to aggregate during solidification in planes held together by strong mutual attractions, whilst the weaker fields on the surfaces hold the residual water. B. P. RIDGE.

Use of starch in paper. WREDE.—See V.

PATENTS.

Improving the colour and purity of juices extracted from sugar beet. A. W. F. CAPPS (B.P. 291,866, 9.3.27).—In the desiccation process of beet sugar manufacture the cossettes, before being dried, are treated to remove the surface sap from the cut cells, e.g., by rapid washing with a sugar solution, or by steaming in such a way that the surface sap, diluted by condensed steam, is blown away from the cossettes. The relatively impure thin juice thus obtained can be limed and carbonated. The cossettes, after drying, yield thick juices of improved quality. J. H. LANE.

Manufacture of dextrose. E. C. R. MARKS. From CORN PRODUCTS REFINING CO. (B.P. 291,991, 19.8.27).—The conversion of starch with acid is interrupted when about 40—42% of sugar has been formed, and the solution is neutralised to p_H 4.5—4.8, filtered, adjusted to p_H 5.5—6.5, concentrated to d 1.2—1.26, and again filtered through bone char. After dilution and re-acidification the conversion is completed, preferably in a glass enamel-lined converter. The filtrations, by removing impurities, especially traces of heavy-metal salts, prevent to a large extent the formation of gentiobiose and other condensation products liable otherwise to be formed during the later stages of saccharification. Yields of 95—98% of dextrose can be obtained.

J. H. LANE.

Lævulose from purified dahlia juice. Lævulose from soluble non-sugar carbohydrates of the dahlia. W. C. ARSEM, Assr. to INDUSTRIAL TECHNICS CORP. (U.S.P. 1,663,233—4, 20.3.28. Appl., 31.1.27).—The juice is clarified and the mixture of inulin and non-sugar carbohydrates is (A) hydrolysed by boiling with sufficient tartaric acid to convert the whole of the carbohydrates into lævulose, or (B) treated for separation of the inulin, and the remaining carbohydrates are hydrolysed to lævulose. After cooling, protein impurities are removed by fermentation with pepsin etc., the solution is concentrated, and the lævulose separated by seeding etc. F. R. ENNOS.

XVIII.—FERMENTATION INDUSTRIES.

Determination of the fusel oil content of brandy by Röse's method. B. LAMPE and W. KILP (Z. Spiritus-ind., 1928, 51, 199—200).—The method is based on the fact that alcohol containing fusel oil when extracted with chloroform causes a greater volume increase of the chloroform than pure alcohol. The alcohol content of the brandy is first determined and the concentration of the alcohol adjusted to 24.68% by weight, either by means of distilled water free from carbon dioxide or by absolute alcohol, according as the concentration is over or under that figure. If necessary, any carbon dioxide present in the original spirit should be expelled by distillation under a reflux condenser. The Röse burette is immersed in a water bath at 15° and filled to the 20 c.c. mark with chloroform by a drawn funnel so that the walls of the burette are not wet with the liquid. 100 c.c. of the spirit of 24.68% concentration with 1 c.c. of sulphuric acid of d 1.286 are added, and the mixture, after being thoroughly shaken, is kept at 15° for about $\frac{1}{2}$ hr., no drops of chloroform being allowed to remain in the upper part of the burette. From the volume of chloroform so obtained, and from that found by repeating the measurement with pure brandy, the weight of fusel oil in 100 g. of original spirit is calculated by multiplying the difference between the two volumes of chloroform in c.c. by 2.2. For spirit with a high content of fusel oil, a modification is necessary owing to the separation of the oil on dilution with water. According to the expected oil content, 10, 20, or 30 c.c. of the spirit are mixed with 75 c.c. of spirit free from fusel oil, and subsequently diluted with distilled water free from carbon dioxide, so that 24.68% by weight of alcohol is present. After making a correction for loss of liquid by adding 1 c.c. to the end-volume, the method is as described above. C. RANKEN.

Peptone. GROLL.—See XX.

PATENTS.

Manufacture of acetone and butyl alcohol [by fermentation]. G. W. FREIBERG (U.S.P. 1,672,487, 5.6.28. Appl., 29.12.22).—The removal of calcium and magnesium compounds by means of water-softeners (particularly zeolites) from the water used in the fermentation process decreases the formation of acids and so increases the yield of acetone and butyl alcohol.

C. HOLLINS.

Manufacture of yeast, especially by the aeration process. A. POLLAK (U.S.P. 1,677,529, 17.7.28. Appl., 23.3.23. Austria, 4.4.22).—See B.P. 195,963; B., 1924, 687.

XIX.—FOODS.

Cacao tannin and its determination. H. R. JENSEN (Analyst, 1928, 53, 365—372).—The tannin is extracted by heating 25 g. of normal, fully roasted, unalkalised cacao nib with 460 c.c. of water mixed with about 37 c.c. of 0.1N-sodium hydroxide. Highest results were obtained after neutralisation of the natural free acidity of the nib. It is sometimes necessary to remove theobromine by chloroform before precipitation. The water-soluble tannin content for eleven samples of full-roast nib was 5.2—6.5%, equivalent to an average tannin content of 13.8% on the fat-free and dry cacao.

Fine mild Ceylon cacao (Criollo) did not contain less tannin than the stronger Accra and Trinidad cacaos (Forastero), and the highest recorded value was obtained from a sample of chocolate, perhaps as a result of alkalisatation before roasting decreasing the conversion of tannin into phlobaphen. A mixed cacao husk contained 8.76% of tannin. The average nitrogen content of the precipitates prepared by the standard alkalisatation was 4.49%, similar to the value (4.3%) for hop and tea tannin compounds. D. G. HEWER.

Properties of cacao butter and detection of foreign fats in chocolate. D. ALBERS (Chem. Weekblad, 1928, 25, 235—239).—The separation temperature of a mixture of 1 pt. of cacao butter with 4 pts. of aniline varies with the acidity of the fat; with the pure neutralised fat, the separation temperature should be at least 42°. The m.p. varies according to the conditions; there appear to be at least three modifications. None of the ordinary constants is of much help for detection of adulterants, unless these are present in considerable amounts; the most useful are the separation temperature, having regard to the acidity, the refractive index, and the m.p., determined with regard to the behaviour under different conditions.

S. I. LEVY.

PATENTS.

Flour-treating process. F. L. DUNLAP, Assr. to INDUSTRIAL APPLIANCE Co. (U.S.P. 1,661,250, 6.3.28. 16.11.23).—Wheat flour is treated with sufficient alkali to give it a p_H value of 9—12; a shortening effect in the baked products is thus produced. F. R. ENNOS.

Preparation of dough. H. C. HERENDEEN (U.S.P. 1,660,839, 28.2.28. Appl., 20.9.26).—During the preparation of dough the flour is stirred in contact with steam. F. R. ENNOS.

Preparation of a powdered milk for ice-cream mix. A. S. COSLER, Assr. to TURNEY PROCESSES, INC. (U.S.P. 1,659,723, 21.2.28. Appl., 25.4.27).—Skim milk is thickened by heating without scorching above the coagulating point of lactalbumin (80—100°), and is then treated with an alkaline solution of lime and sugar, mixed, and dried to a powder. F. R. ENNOS.

Manufacture of soft cheese. A. C. DAHLBERG, Assr. to U.S.A. (U.S.P. 1,661,601, 6.3.28. Appl., 3.8.27).—A mixture of sweet cream with 5% of soluble, dry, skim milk and $\frac{1}{2}$ % of powdered agar is pasteurised and cooled. After adding salt and a starter, the mixture is strained, homogenised, and cooled to 21°, at which temperature it is maintained until the cheese has developed a mild acid flavour. F. R. ENNOS.

Treating oils etc. (B.P. 293,255). **Fats from animal matter etc.** (B.P. 292,327 and 292,719).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Examination of colloidal silver preparations. I. M. KOLTHOFF (Pharm. Weekblad, 1928, 65, 362—365).—The method of detecting ionic silver by precipitating the colloidal matter by means of magnesium sulphate or alum and adding a chloride to the clear filtrate is not always applicable, since the removal of the colloids is

often incomplete. The employment of an ultrafiltration method is suggested.

S. I. LEVY.

Metal content of homœopathic iron powders. A. KUHN (Pharm. Ztg., 1928, 73, 827—828).—The variations in composition of powders prepared from ferrum reductum and lactose are ascribed partly to the varying content of metal in the iron, and partly to the possibility that the purer, softer metal may be forced into the walls of the iron mortar by long trituration. The metal content may be determined, in the stronger preparations, by igniting, dissolving the ash, reducing with zinc, and titrating with permanganate. For weaker preparations, a colorimetric method based on the use of ferrocyanide is recommended. For the weakest, a colorimetric method employing thiocyanate, in which the coloured solution is extracted with ether for the comparison, is the most suitable.

S. I. LEVY.

Preparation of peptone for therapeutic applications. J. T. GROLL (Pharm. Weekblad, 1928, 65, 465—474).—A method of obtaining completely soluble peptones of good taste from casein, beef, and blood fibrin by fermentation with pepsin in acid solution, followed by fermentation with trypsin in alkaline solution, filtration, and evaporation in vacuum, is described. The formaldehyde titration of these preparations shows a uniform high degree of hydrolysis of the protein, but the ash content, mostly sodium chloride formed from the employment of sodium hydroxide and hydrochloric acid to secure the desired degrees of alkalinity and acidity for the fermentations respectively, is high. The bitter taste and irregular pharmacological effect of some commercial peptones is ascribed to the selection of acid hydrolysis methods for the preparation.

S. I. LEVY.

Citronellal and citronella oils. H. I. WATERMAN and E. B. ELSBACH (Rec. trav. chim., 1928, 47, 764—775).—The increase in refractive index and sp. gr. previously observed (B., 1927, 669) on keeping citronellal in contact with air has been confirmed in the case of Java and Ceylon citronella oils. Of three samples of commercial citronellal, (A) n_D^{20} 1.4551, d_4^{20} 0.8706, (B) n_D^{20} 1.46326, d_4^{20} 0.9064° (purified by the bisulphite method), (C) n_D^{20} 1.44887, d_4^{20} 0.8573, only (C), prepared by fractionation of Java citronella oil, proved reasonably pure on vacuum distillation. In all fractions from these oils high refractivity and sp. gr. are parallel, and their inferiority is attributed to absorption of atmospheric oxygen. The fractions obtained by distillation of sample (C) all showed slight increases in refractivity and sp. gr., accompanied by an absorption of oxygen, on keeping for three months in contact with air. The deterioration can be prevented partly or completely not only by exclusion of air but by the addition of traces of quinol or of 7—17% of geraniol. The high refractivity of "oxidised" citronellal is not entirely due to the presence of the citronellic acid formed, and the absorption of oxygen is in excess of that required for the formation of the citronellic acid found. The stability of citronella oil, in comparison with citronellal, is probably due to the presence of geraniol.

R. BRIGHTMAN.

Determination of alcohols in ethereal oils. I. G.

OSBERHARD and N. A. KUBASSOV (Pharm. Ztg., 1928, 73, 839—841).—The formulæ given in the various handbooks for calculating the alcohol content from the ester and acetyl values of ethereal oils are found to be unsatisfactory, and the following formula has been calculated: $A = Ma(n - 0.021b)/20n(s - 0.021a)$, where A is the total content of alcohol (mol. wt. M), n = wt. of oil taken, b = c.c. of 0.5*N*-alcoholic potash for saponifying esters in n g. of oil, s = wt. of acetylated oil, and a = c.c. of 0.5*N*-alcoholic potash for saponifying esters in s g. of acetylated oil. Results obtained with synthetic mixtures of such alcohols as menthol, thymol, and borneol with high-boiling paraffin or freshly-distilled pinene were in agreement with the formula. For complete acetylation, oils containing menthol require at least 75 min., those containing thymol or borneol at least 105 min.

S. I. LEVY.

Balsams and storax. ROSENTHALER.—See XIII.

PATENTS.

Preparation of *p*-alkoxyphenylglycinamides. J. ROSIN, Assr. to MERCK & Co. (U.S.P. 1,672,689, 5.6.28. Appl., 22.7.26).—*p*-Alkoxyphenylglycinamides, substantially non-toxic antipyretic or analgesic substances, are prepared by condensing *p*-aminophenol ethers with chloroacetamide (or with ethyl chloroacetate, followed by amidation). *p*-Ethoxyphenylglycinamide, m.p. 142—145°, is described.

C. HOLLINS.

Production of alkyl mercuric sulphur compounds. M. S. KHARASCH (U.S.P. 1,672,615, 5.6.28. Appl., 29.6.27).—Soluble compounds of the type, $R \cdot Hg \cdot S \cdot R'$, in which R' is an alkyl group (preferably methyl or ethyl) and R carries a carboxylic or sulphonic group, are obtained by the interaction of an alkyl mercuri-chloride or -hydroxide in alcoholic suspension with a carboxylic or sulphonic acid containing a thiol group. The following compounds are prepared:— $R \cdot Hg \cdot S \cdot CH_2 \cdot CO_2H$, $R = Me$ (m.p. 87°), Et (m.p. 79°), Pr (m.p. 73°), $n-Bu$ (m.p. 68°); $R \cdot Hg \cdot S \cdot CH_2 \cdot CH_2 \cdot CO_2H$, $R = Et$, $n-Bu$, *isoamyl* (m.p. 93°); $R \cdot Hg \cdot S \cdot CHEt \cdot CO_2H$, $R = Me$ (m.p. 73°), Et , $n-Bu$; $R \cdot Hg \cdot S \cdot CH_2 \cdot CH(NH_2) \cdot CO_2H$, $R = Et$, $n-Bu$, *isoamyl* (m.p. 95°); $Et \cdot Hg \cdot S \cdot CH(CO_2H) \cdot CH_2 \cdot CO_2H$; $R \cdot Hg \cdot S \cdot C_6H_4 \cdot CO_2H$, *ortho*, $R = Me$ (m.p. 171°), Et (m.p. 110°), $n-Bu$, *allyl*; *meta*, $R = Et$ (m.p. 105°), $n-Bu$; *para*, $R = Et$, $n-Bu$, *isoamyl*; (5) $R \cdot Hg \cdot S \cdot C_6H_3(OH)(CO_2H)$, $R = Et$, $n-Bu$; $p-Et \cdot Hg \cdot S \cdot C_6H_4 \cdot CH_2 \cdot CO_2H$; $p-R \cdot Hg \cdot S \cdot C_6H_4 \cdot SO_3H$, $R = Me$, Et , *allyl*. The products dissolve in alkali to neutral solutions suitable for intravenous injection.

C. HOLLINS.

Obtaining perfumes from flowers and other parts of plants. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 292,667, and Addn. B.P. 292,668, 21.3.27).—(A) The extraction, etc. is effected below 30° with a solvent of b.p. below 30°, such as ethyl chloride. (B) Air or an indifferent gas is dried and passed over the flowers etc. at such a rate that the latter are not fouled.

B. FULLMAN.

Compound of gall acids. A. GAMS and P. SCHEIDEGGER, Assns. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,677,554, 17.7.28. Appl., 31.7.26. Switz., 2.9.25).—See B.P. 257,900; B., 1927, 572.

Unsaturated aldehydes (B.P. 284,458).—See III.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Fibrous structure of silver bromide in the films of photographic dry plates. H. H. MEYER (Ann. Physik, 1928, [iv], 86, 325—352).—X-Ray photographic investigation of the silver bromide in the films of photographic dry plates shows that that compound exhibits a fibrous structure. As the gelatin film dries the octahedral (111) planes tend to arrange themselves parallel to the film. The distributions of the (100), (110), and (111) planes relative to difference angles of incidence have been quantitatively determined and the data employed for a qualitative explanation of the difference between the X-ray diagrams obtained with silver bromide without binding material and that in photographic films. Pure gelatin, dried in a film on a solid support, also shows a pronounced crystal diagram.

J. W. SMITH.

Optical sensitisation of silver iodide. K. BAUKLOH (Z. wiss. Phot., 1928, 25, 233—262).—Contrary to the statements of Lüppo-Cramer, silver iodide can be sensitised. Pure silver iodide layers on a silver support, similar to Daguerrotype plates, can be optically sensitised by bathing in various dyes (erythrosin, pinacyanol, phenosafranine). The sensitivity thus obtained is fairly uniformly distributed over the whole spectrum. With such plates the maximum sensitivity occurs when a loop of the stationary light wave lies in the surface of the silver bromide layer, as had previously been suggested for Daguerrotype plates by Wiener and Scholl (Wied. Ann., 1899, 68, 145, 150). Silver iodide-gelatin layers can be sensitised in the same way. As far as susceptibility to sensitising is concerned there is no fundamental difference between medium-free and gelatin-silver iodide layers. Certain non-sensitised silver bromide-gelatin plates which had been kept for two years showed, on being converted into silver iodide, a sensitivity extending into the extreme red, even without sensitising by dyes. This was found to be due to the action of mercury vapour. Even before conversion into iodide these plates were slightly sensitised. Mercury is thus a sensitiser for silver bromide and silver iodide.

W. CLARK.

Nature of desensitisation. E. BAUR (Z. wiss. Phot., 1928, 25, 263—264).—Silhonen's explanation (cf. B., 1928, 69) of Kögel and Steigmann's theory of desensitisation (B., 1926, 386) is improved.

W. CLARK.

Solarisation of silver bromide. LÜPPO-CRAMER (Z. wiss. Phot., 1928, 25, 224—229).—Curves are given showing the solarising properties of a number of commercial photographic emulsions. Different plates show extraordinarily differing shapes of the curve in the region of solarisation. Curves also show the effects of a number of bromine acceptors in reducing solarisation. Even in the case of plates which readily show solarisation, the solarisation sets in long before any direct blackening can be observed. In the case of plates bathed in bromine acceptors there is only a very slight direct blackening resulting from solarising exposures. Determinations of the silver formed on exposure have no appreciable significance for the study of the latent image, because silver can only be obtained in quantities sufficient for determination if

exposures falling in the solarisation region are given. Values obtained cannot be extrapolated for the latent image if solarisation consists essentially of a reversal of the normal photochemical process of the decomposition of silver bromide.

W. CLARK.

PATENTS.

Non-inflammable photographic films. L. WESTON (B.P. 293,269, 5.1.28).—To prevent non-inflammable films from crinkling and shrinkage during development and fixing, the film base, before coating with the emulsion, is drawn slowly through water, preferably at 30—50° for 12 to 48 hrs., the adherent water being removed mechanically before drying. The process can be accelerated by the addition of a small amount of ammonia or an amino- or amido-compound such as aniline, acetamide, urea, etc. to the water.

J. W. GLASSETT.

Colour photography or kinematography. Soc. DU FILM EN COULEURS KELLER-DORIAN, and P. A. RICHARD (B.P. 263,115, 18.11.26. Fr., 19.12.25).

XXII.—EXPLOSIVES; MATCHES.

Abel test. I and II. KOEHLER and MARQUEYROL (Mém. Poudres, 1928, 23, 11—17, 17—18).—I. Experiments with guncotton show that calcium carbonate, present in all French nitrocottons used for military purposes, exerts a greater influence in lowering the values obtained in the Abel test when the moisture present before drying increases. This lowering is caused by the formation of calcium nitrite by the interaction in presence of water of calcium carbonate and nitrocellulose. It is concluded that the Abel test measures anything rather than the stability of the powder and its replacement as an official test by the Bergmann-Junk test is recommended. If, for any reason, it cannot be abolished certain modifications of it are described.

II. Experiments on a soluble nitrocellulose (CP₂) lead to the same conclusion.

S. BINNING.

Determination of alcohol, ether, and water in the solvent vapours eliminated during the drying of powders. L. A. CHENEL (Mém. Poudres, 1928, 23, 4—6).—Caustic potash removes completely the vapours of ethyl and amyl alcohols when a stream of air containing them is passed through it and allows any ether vapour to pass unabsorbed. Some of the alcohol, however, is oxidised, which renders the method useless for quantitative work, but if the air is replaced by nitrogen accurate results are obtained.

S. BINNING.

Distribution of diphenylamine in B powder. J. DESMAROUX (Mém. Poudres, 1928, 23, 33—35).—A certain amount of the diphenylamine introduced into B powder during its incorporation is lost in subsequent processes during manufacture. Examination of the surface and interior of the individual units of B powder showed a concentration of diphenylamine in the superficial layers. It appears that this segregation takes place before the secondary drying after the water steeping. During the primary drying the solvent in the interior of the powder carries with it diphenylamine in solution which is deposited in the superficial layers when the solvent evaporates.

S. BINNING.

Determination of organic matter in waste mixed

acid and of carbon in nitrocellulose. CARRIÈRE and GUIBERT (Mém. Poudres, 1928, 23, 19—28).—A solution of chromic anhydride in sulphuric acid may be used for determining organic matter in waste mixed acid and also of carbon in nitrocellulose. In both cases, the carbon is oxidised to carbon dioxide, which is absorbed in a solution of caustic potash. The percentage of carbon in guncotton (CP₁) is found to be 25.45 and in soluble nitrocellulose (CP₂) to be 28.32. CP₁ is therefore practically cellulose endecanitate, and CP₂ a nonanitic ester of a monoxide or dioxide of cellulose. S. BINNING.

Fixation of camphor by nitrocellulose. DESMAROUX (Mém. Poudres, 1928, 23, 54—62).—The fixation of camphor by nitrocellulose was investigated by dissolving camphor in a liquid saturated hydrocarbon and measuring the rate at which camphor was taken up by nitrocellulose steeped in the solution. This rate was determined from the change in the refractive index of the camphor-hydrocarbon solution at various times. The absorption curves show the existence of a critical concentration below which camphor is not taken up by nitrocellulose. Other experiments based on the rate at which camphor evaporates from a film of nitrocellulose gelatinised by camphor showed a discontinuity in the vapour pressure when a certain critical concentration was reached. A complex combination between camphor and nitrocellulose probably exists, the vapour pressure of which is much lower than that of a solution of free camphor of the same concentration. With nitrocelluloses of various nitrogen contents, the critical concentration of camphor increases with increasing nitrogen content. S. BINNING.

Products formed during storage of SD powder. LÉCORCHÉ and JOVINET (Mém. Poudres, 1928, 23, 69—78).—SD powder contains soluble nitrocellulose 66, nitroglycerin 27, and centralite (*s*-diphenyldiethylcarbamide) 7%. A sample that had been stored at 50° in a dry atmosphere for four years was dissolved in lye and distilled in steam, when centralite and a compound considered to be phenylethylnitrosoamine were found in the distillate. It is supposed that this compound is formed by hydrolysis of centralite by nitrous and nitric acids formed by the decomposition of nitrocellulose and nitroglycerin. In the powder a further change to nitrosodinitroethylaniline takes place. Analytical methods are given for determining centralite and phenylethylnitrosoamine in powders containing nitroglycerin and nitrocellulose. S. BINNING.

Products of conversion of amyl alcohol in BAM powders. MARQUEYROL (Mém. Poudres, 1928, 23, 7—10).—Investigation of BAM powders (nitrocellulose powders stabilised with amyl alcohol) heated at 75° show the presence of amyl nitrite and certain of its normal decomposition products such as valeric acid, amyl valerate, and probably in presence of air, amyl nitrate. When amyl alcohol is present, amyl nitrite is probably the primary decomposition product of nitrocellulose. S. BINNING.

Determination of camphor in [propellant] powders. DESMAROUX (Mém. Poudres, 1928, 23, 43—53).—Camphor can be determined in propellant powders by

dissolving the powder in lye, removing the camphor by distillation in steam, dissolving it in water, and determining the surface tension of the solution by the drop method. The capillary used for the formation of the drops should be standardised with camphor solutions of known strength. Any nitroglycerin or diphenylamine present in the powder does not affect the results, but substituted carbamides render the method useless in spite of their low solubility in water.

S. BINNING.

Determination of nitrites in nitrocellulose. KOEHLER and MARQUEYROL (Mém. Poudres, 1928, 23, 19—28).—The nitrocellulose is extracted with either hot or cold water, and the extract compared colorimetrically by means of Griess' reagent with solutions of sodium nitrite of known strengths. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Volatile acids in digesting sewage sludge. H. HEUKELEKIAN (Ind. Eng. Chem., 1928, 20, 752—755).—The determination of volatile acids by steam distillation in digesting sewage sludge shows that in unseeded and unlimed material they reach a maximum shortly after the digestion has started and persist for the whole digestion period. In limed, unseeded sludge the total volatile acidity determined after acidifying with sulphuric acid reaches a maximum early in the digestion and then decreases very rapidly so that within 100 days when digestion is complete they are at a low level. There is an intimate relation between volatile acids and gas production, and volatile acid determinations can be taken as an additional index of the completion of digestion in conjunction with gas measurements, biochemical oxygen demand, and solids and ash determinations; when the digestion is complete as indicated by the usual methods volatile acids have practically disappeared.

W. G. CAREY.

Apparatus for micro-determination of dissolved oxygen [in water]. T. G. THOMPSON and A. C. MILLER (Ind. Eng. Chem., 1928, 20, 774).—The sample tube and its connexions hold 5—10 c.c. of water, which is the quantity used for the determination. Capillary U-tubes which connect with the reservoir of water to be tested also serve for holding the quantity of water (0.02 c.c.) displaced by the successive addition of manganese sulphate, alkaline potassium iodide, and sulphuric acid from three graduated capillaries. The final solution is washed out and titrated with 0.002N-sodium thiosulphate. C. IRWIN.

Determination of oxygen in water. F. LIEBERT and W. M. DEERNS (Chem. Weekblad, 1928, 25, 226—228).—Of the many methods examined, that of Winkler (cf. B., 1915, 679) alone gave consistently accurate results, provided organic matter was not too high.

S. I. LEVY.

Factors affecting toxicity of hydrocyanic acid to insects. F. J. BRINLEY and R. H. BAKER (Biol. Bull., 1927, 53, 201—207).—Within certain limits concentration and time of exposure to hydrocyanic acid are inversely related in regard to toxicity to insects. Toxicity increased with rising temperature, but the degree of humidity in the atmosphere did not appear to be important. C. T. GIMMINGHAM.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPT. 14, 1928.

I.—GENERAL; PLANT; MACHINERY.

Value of inert gas as a preventive of dust explosions in grinding equipment. H. R. BROWN (U.S. Dept. Agric. Tech. Bull. 74, June, 1928, 23 pp.).—Results of experiments in grinding oat hulls on a scale comparable with industrial conditions and in a plant so designed that the atmosphere might be controlled by the admission of carbon dioxide (boiler flue gas) showed that no ignition of dust occurred in the mill when the oxygen content of the atmosphere was below 15%, the ignition agent being an electric arc. Under varying industrial conditions of grinding a 12% oxygen content is considered completely safe. The use of an inert gas, *e.g.*, carbon dioxide, is considered valuable not only in preventing dust explosions wherever an explosion hazard exists, but also for fire-extinguishing purposes, since no injury is caused to plant or materials as is usually experienced with other methods of fire extinction. C. A. KING.

Method of evaluation of analyses. I. O. LIESCHIE (Z. angew. Chem., 1928, 41, 748—750).—A systematic method is described for the calculation of the composition of a complicated mixture, as regards the actual simple, double, or complex salts present, from the usual analytical data giving the percentages of anions and cations or basic and acidic oxides. Examples of the application of the method to analyses of ultramarine and of a hard water are given.

A. DAVIDSON.

The unimeter, a new [optical] instrument for chemical industry. L. BLOCH and H. G. FRÜHLING (Chem.-Ztg., 1928, 52, 488—490).—By means of a polarising photometer fixed to a movable arm, the intensity and colour of the reflected or transmitted light from solids, liquids, or solutions are compared with standards, the intensity of the light being proportional to the square of the tangent of the angle of rotation of the photometer head. The instrument may also be adapted for the examination of large or of distant objects, so that the course of a chemical or physical change may be followed and controlled by the variation in the optical behaviour of the materials.

F. R. ENNOS.

Separation of fractions and the size and shape of rectification columns. K. V. KOSTRIN (Nef. Choz., 1927, 13, 180—187).—The fuel consumption of stills is increased by lengthening the column, but not always by increasing its cross-section. The efficiencies of rectifying columns are compared.

CHEMICAL ABSTRACTS.

Changes in construction of modern stills.

S. VUISHETRAVSKI (Azerbeidj. Nef. Choz., 1928, No. 3, 58—69).—Two new Russian stills are described. CHEMICAL ABSTRACTS.

Tube stills. ACKERMAN.—See II.

PATENTS.

Tunnel kilns. A. HERMANSEN (B.P. 292,781, 7.7.27).—A tunnel kiln of the duplex type in which two lines of traffic move in opposite directions and in an intermittent manner, with heat exchange transversely, is provided with transverse partitions combined with the trucks, two such partitions and a pillar of a central row of fixed pillars being in line during the intervals of rest of the trucks. B. M. VENABLES.

Heat-treating apparatus. C. L. IPSEN, ASST. to GEN. ELECTRIC CO. (U.S.P. 1,675,271, 26.6.28. Appl., 29.12.26).—The material is placed in a number of receptacles which are passed through a long furnace, and the contents of each are dumped as they pass over a discharge opening. B. M. VENABLES.

Set pans and the like. I. G. FARBERIND. A.-G. (B.P. 284,281, 17.1.28. Ger., 27.1.27).—A pan is constructed with Perkins heating or cooling tubes cast on the outer surface of, or buried in the middle of, the wall of a cast-iron pan which is enamelled.

B. M. VENABLES.

Grinding mills. W. F. CARR-HILL (B.P. 293,559, 23.6.27).—A grinding mill of the centrifugal roll type is provided with resilient suspending and driving means for the roll shafts, so that they can move slightly independently of their driving gear, such movement increasing the relative rubbing between the rolls and ring. B. M. VENABLES.

Grinding mill. J. MURSCHE, ASST. to BUFFALO HAMMER MILL CORP. (U.S.P. 1,675,658, 3.7.28. Appl., 20.6.27).—In a disintegrator provided with a driving boss and pairs of swinging links, every hammer head is arranged with several working faces, has a circular concentric hole, and is keyed positively in any one of a number of positions on a short shaft which is eccentrically and loosely pivoted between a pair of swinging links, so that one desired grinding face is always presented to the material, but not rigidly. B. M. VENABLES.

Machine for crushing ore, stone, etc. J. W. ERICSSON (B.P. 293,651, 18.1.28).—The machine resembles a horizontal disc crusher, but the moving disc is reciprocated axially and not gyrated; the reciprocating motion is produced by the eccentric gyration of a toggle bar, and arrangements are made to keep the crushing strains as self-contained as possible. B. M. VENABLES.

Disintegrating, grinding, reducing to fibres, or mixing moist or dry materials. E. STERZL

(B.P. 276,356, 19.8.27. Ger., 20.8.26).—In apparatus wherein rings of teeth on a rotating cylinder run between rings of teeth on the interior of the casing, the faces of the teeth are inclined (*e.g.*, at 23° to the axial plane) so as to produce longitudinal movement of the material under treatment, both ways from the inlet if that is at the centre, or in one direction if the inlet is at one end of the apparatus. B. M. VENABLES.

Crushing apparatus. E. B. SYMONS (B.P. 293,642, 23.12.27).—Further improvements are claimed in the gyratory cone crusher described in B.P. 243,926 (B., 1926, 111). B. M. VENABLES.

Apparatus for separating materials of different density. J. W. HARTLEY (B.P. 293,137, 4.4.27).—In apparatus of the type where a bucket elevator or raff wheel removes lighter material floating on water and an endless conveyor material which sinks, an upward current of water is maintained in front of the raff wheel by circulating pumps or propellers which draw the upward current from behind the raff wheel, enabling material which would otherwise just sink to be caught. Mechanical improvements are described. B. M. VENABLES.

Apparatus for the continuous drying or distillation of fine granular masses. TROCKNUNGS-, VERSCHWELUNGS-, & VERGASUNGS-GES.M.B.H., and L. HONIGMANN (B.P. 293,147, 22.4.27).—The granular material passes through a cylindrical chamber which is traversed also by a gaseous heating medium, using as heat-exchanging devices a number of rotating or oscillating perforated metal discs attached to a central driving shaft. When it is desired to collect the volatile products of distillation, the discs do not come into direct contact with the heating gases in the upper part of the container, but are heated by radiation from fixed heating bodies interposed between them. In another form of apparatus the drum itself may rotate and the heat-exchange bodies may be rigidly attached to it. A. B. MANNING.

Effecting reactions between solid materials and air or gas. E. HAYWARD (B.P. 292,642, 22.2.27).—An apparatus by which pulverulent, dry material may be continuously treated in a counter-current of gas at raised temperatures and pressures, say 450° and 20 atm., comprises a closed vessel with internal worm or other conveyor, and external heating. The solid is admitted and discharged in regular cycles by means of valves from or to intermediate chambers at the ends, the necessary valves being automatically operated in correct sequence. B. M. VENABLES.

Apparatus for the production of dispersions of solids or liquids. W. H. WHATMOUGH (B.P. 293,510, 29.4.27).—The apparatus is specially suitable for breaking down globular, amorphous aggregates produced by mills of the Plauson or Premier type and referred to in B.P. 276,400 (B., 1927, 831), and consists of a high-speed rotor covered with mesh work through which the liquid is forced. B. M. VENABLES.

Rotary vacuum filters etc. BRINJES & GOODWIN, LTD., R. STEWART, and C. F. N. SEAMAN (B.P. 292,644, 24.2.27).—A form of rotary drum filter is described, having provision for separate withdrawal of filtrate and washings. B. M. VENABLES.

Method and apparatus for filtering air. REED AIR FILTER CO., ASSEES. OF C. P. HEGAN (B.P. 279,102, 14.10.27. U.S., 15.10.26).—The dust is caught on surfaces coated with a viscous liquid such as oil, which when fully charged with dust is renewed, without heating, by a large flush of new liquid, the flushings being filtered and re-used. The flushing is done when the air current is cut off, and automatic switches may be used which operate so that when the air fan or air current stops, the motor for the oil pump starts. B. M. VENABLES.

Filter. E. J. SWEETLAND (U.S.P. 1,677,503, 17.7.28. Appl., 22.1.20).—The end portion of a strip of filtering fabric lies flat upon the bottom of a one-piece casing, and the remainder of the strip is folded backwards and forwards upon itself, spacing members being placed between each fold, until the casing is full. The lateral free edges of the fabric are folded within channels on opposite sides of the casing in such a manner that solids are prevented from escaping from the folds without sewing or cutting the fabric. F. G. CLARKE.

Discharge of liquids from centrifugal separators. AKTIEBOLAGET SEPARATOR (B.P. 285,088, 9.2.28. Swed., 12.2.27).—In a centrifugal separator for two liquids the heavier liquid is led, prior to discharging, to a point close to the axis and preferably above the outlet for lighter liquid, and the passages are arranged so as to be easily cleaned. B. M. VENABLES.

Liquid atomisers. SIEMENS-SCHUCKERTWERKE A.-G., ASSEES. OF SIEMENS-SCHUCKERTWERKE GES.M.B.H. (B.P. 287,105, 27.2.28, and 292,940, 27.3.28. Ger., 15.3. and 27.6.27).—A form of spray especially suitable for humidifying purposes comprising a specially shaped disc rotated at very high speed by an electric motor with vertical shaft. B. M. VENABLES.

Production of liquid dispersions. ANODE RUBBER CO., LTD. From P. KLEIN and A. SZEGVÁRI (B.P. 293,071, 1.3.27).—The mixture is subjected to the action of hard, rounded, grinding elements which are kept in motion by a stirring device, and is preferably circulated between the emulsifier and a sedimentary or centrifugal separator in which the coarser unemulsified particles are separated. The size of the grinding elements should lie between 1 mm. and 8 mm. mesh, and two sizes may be used, one being at least double the diameter of the other. The metallic parts of the apparatus may be covered with rubber, and cooling may be applied. B. M. VENABLES.

Circulating or mixing of liquids etc. DUNLOP RUBBER CO., LTD., R. F. MCKAY, H. WILLSHAW, W. G. GORHAM, and R. F. LEE (B.P. 293,072, 2.3.27).—A bath of liquid matrix, such as rubber ready for deposition on moulds, is kept in circulation by pumping, the delivery of the pump being upwards and fan-shaped at the centre of the bath, and the flow then along the surface to weirs at the ends. Any coagulated matter is caught in strainers at the weirs and the overflow liquid runs into a sump (where make-up is added) and returns to the pump. The weirs also prevent increase in depth of the bath due to the presence of extra large moulds. B. M. VENABLES.

Evaporators. G. & J. WEIR, LTD., and J. SIM (B.P. 292,842, 22.11.27).—The liquid in the evaporator

is subdivided by trays into two or more bodies each having a free evaporating surface, and each heated by steam tubes through which the steam passes in parallel. The level of the liquid in the lowest chamber, which is the bottom of the containing shell, controls by a float valve the feed of liquid, which is wholly to the uppermost chamber, the liquid overflowing downwards from tray to tray.

B. M. VENABLES.

Continuous treatment [e.g., hydrogenation] of liquids with gases. H. MIELCK (B.P. 272,913, 14.6.27. Ger., 15.6.26).—A long, horizontal, stationary autoclave is divided by moderately permeable partitions into a number of separate chambers, which may be separately heated or cooled, and all are provided with stirrers preferably fixed on a long common shaft. The permeability of the partitions is sufficiently low to prevent any mixing of liquids in adjacent compartments, while permitting only a slow one-way flow.

B. M. VENABLES.

Apparatus for rarefying gases. J. O. BOVING (B.P. 292,758, 31.5.27).—In an apparatus where liquid flows in a siphon tube, a vacuum being produced at the top of the siphon, the delivery branch is arranged on the Venturi principle with high velocity at the top and low at the bottom, thus reducing the height of the necessary liquid column. The throat is also smaller than the inlet conduit, and the gas inlet or aspirator connexion from the vessel to be exhausted is situated in the midst of the stream at or just after the highest point, and is given a stream-line form with a number of small holes.

B. M. VENABLES.

Gas diffuser. E. J. SWEETLAND (U.S.P. 1,677,502, 17.7.28. Appl., 19.12.19).—The porous blocks are treated with a preservative which does not obstruct the pores.

F. G. CLARKE.

Liquefaction apparatus. R. C. TOLMAN, W. L. DE BAUFRE, J. W. DAVIS, and M. H. ROBERTS, ASSTS. to S. G. ALLEN (U.S.P. 1,676,225, 3.7.28. Appl., 16.9.22).—The gaseous mixture is pre-cooled in heat exchangers and passes on to a liquefier for the greater part of the undesired constituent of the gas, and to a separating chamber whence the gas passes to a purifier cooled by a separate refrigerating cycle. The liquid from the separator and the purifier is used to cool (by counter-current) the liquefier and heat exchangers.

B. M. VENABLES.

Production and utilisation of cold. G. CLAUDE and P. BOUCHEROT (B.P. 278,762, 11.10.27. Fr., 11.10.26).—Cold water is drawn through a conduit, which may be heat-insulated, from the depths of the ocean, and the cold water either taken directly to the place where cold is required or used to cool and dry air in a heat interchanger or spray device. At a depth of 1000 m. the temperature of the sea is about 4° even near the equator.

B. M. VENABLES.

Refrigerating compounds. O. HANNACH (B.P. 293,581, 4.8.27).—A mixture, e.g., containing 2 pts. of ammonium chloride and 3 pts. of sodium carbonate, added to 6 pts. of water gives a maximum decrease of temperature (31°).

H. ROYAL-DAWSON.

Carrying out exothermic catalytic chemical reactions. L'AIR LIQUIDE SOC. ANON. POUR L'ÉTUDE

ET L'EXPLOIT. DES PROC. G. CLAUDE, Assees. of SOC. CHIM. DE LA GRANDE PAROISSE (AZOTE ET PROD. CHIM.) (B.P. 289,759, 18.7.27. Fr., 30.4.27).—In catalytic reactions such as removal of carbon monoxide from a mixture of nitrogen and hydrogen by combining the first with part of the last, it is usual to provide heat interchange between the catalytic material and the incoming gas. In order to obtain regulation of the heating the stream of gas is divided into two streams, separately regulable either at the inlet or outlet, which are passed over different surfaces for heat interchange.

B. M. VENABLES.

Absorbing agent. A. ROSENHEIM (B.P. 275,203, 12.7.27. Ger., 30.7.26).—Natural or artificial zeolites or similar substances (e.g., the reaction products of silicates and aluminates or iron compounds etc.) are treated with acids or acid salts in the liquid or gaseous state at ordinary or higher temperatures up to the b.p. of the liquids concerned, forming a mixture containing a gel of amphoteric metal compound beside the silicic acid gel. [Stat. ref.]

H. ROYAL-DAWSON.

Colorimeters and analogous instruments. R. A. LEGENDRE (B.P. 285,848, 21.12.27. Belg., 23.2.27).—The sighting system, which makes use of a Pulfrich prism, permits of two separate sighting lines, each of which passes through a unit consisting of three reservoirs or tanks with transparent walls. A unit may be formed by two right-angled triangular prisms placed with their hypotenuses in juxtaposition and a tank of uniform thickness placed against the longer surface of the side of the rectangular prism. It may also be constructed of two co-axial cylindrical tanks in combination with a third of cylindrical shape but not co-axial.

C. RANKEN.

Temperature-regulating system. SELDEN Co., Assees. of C. E. ANDREWS (B.P. 285,499, 18.2.28. U.S., 19.2.27).—See U.S.P. 1,666,251; B., 1928, 391.

Apparatus for boiling or raising the temperature of water and other liquids. C. F. HAMMOND and W. SHACKLETON (U.S.P. 1,675,347, 3.7.28. Appl., 4.9.26. U.K., 5.9.25).—See B.P. 265,253; B., 1927, 240.

Straining or filtering apparatus. F. C. FULCHER and W. R. BELDAM (U.S.P. 1,673,743, 12.6.28. Appl., 12.6.24. U.K., 22.6.23).—See B.P. 223,627; B., 1924, 1000.

Refrigerating system. C. W. VOLLMANN (B.P. 291,856, 7.3.27).

Refrigerating systems. COMSTOCK & WESCOTT, INC., L. F. WHITNEY and E. A. WEAVER (B.P. 292,812, 22.9.27).

Rotary refrigerating apparatus. H. NIELSEN (B.P. 291,835, 25.2.27).

Furnace roofs. E. STRUNK and A. REPPMAN (B.P. 294,026, 12.10.27).

Machines for breaking coal and other friable material. B. NORTON (B.P. 294,068, 12.1.28).

Liquid filters [for fuel oil etc.]. SOC. ANON. DES NOUV. INVENTIONS MÉCANIQUES ET ÉLECTRIQUES (B.P. 278,349, 22.9.27. Fr., 30.9.26).

Thermostats. G. G. ROYER (B.P. 293,930, 3.5.27).

Chemical reactions (B.P. 293,138).—See VII. Muffle kiln (U.S.P. 1,677,818).—See VIII. Rotary kiln (B.P. 293,331).—See IX. Filtering apparatus (B.P. 293,534).—See XXIII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Agglomeration of coals by means of hydrocarbons partially dehydrogenated by sulphur. A. LÉAUTÉ (Compt. rend., 1928, 187, 227—229).—Partial dehydrogenation of the oils used increases their adhesive power, as shown by the increased resistance of the agglomerates to crushing. Briquetting without the addition of pitch is then possible. The effect is less the poorer the specimen in light oils. C. W. GIBBY.

Effects of temperature and pressure on the explosibility of coal dust-air mixtures. H. SANO (Nihon-Kogyokwaishi, 1925, No. 486, 845—852).—The lower limit of inflammability in coal dust-air mixtures descends with rising initial temperature, but is unaffected by change of pressure from 1 to 5 atm., although the ignition temperature at the limit is lowered. Dust clouds incapable of inflaming by ignition can propagate explosions. CHEMICAL ABSTRACTS.

Gas producer operation. V. WINDETT (Proc. Eng. Soc. W. Pa., 1928, 44, 11—34).—The results of a large number of records of the ordinary working of ten Wellman-Seaver-Morgan, Type L, No. 10, mechanical gas producers at a factory of the American Window Glass Co., Jeanette, Pa., are given. The gas is supplied for heating two glass furnaces, each holding 1800 tons of molten glass, and 26 annealing ovens. A curve showing the results from 430 determinations of the heat of combustion of producer gas made from 60,000 tons of coal over 14 months shows a maximum deviation of 0.24%. There is no seasonal effect on the operation of the producers. An inspection of the heat balance indicates loss of heat by radiation is only 4% of the total. The factors chiefly affecting the quantity and quality of the gas are the temperatures of the air blast and of the gas. The results of analyses, based on variations in the temperature of the air blast, show that the gas of highest calorific value is obtained at a saturation temperature of 48.9—54.4°, with a maximum at 51.7° of 174 B.Th.U. net. A tabulation based on gas temperatures shows the most advantageous range is 677—788°. Plottings of the calorific values, or heats of combustion of the gas, and of curves of the principal constituents of the gas shows the existence of a tendency towards an increase in the carbon monoxide, accompanied by a decrease of the carbon dioxide, and also a rise in temperature. The decrease in the hydrogen accounts for the lowering of the calorific value of the gas as the temperature rises. The relationship between the fusing temperature of the ash, the fineness of the coal, and the temperature of the air-blast shows the general desirability of a low percentage of fines and an ash of high fusing point. The total useful heat of gasification is 88.20%, which is the measure of the efficiency of the gas-producer operation. H. S. GARLICK.

Apparatus for determining the ignition point of coke and of technical carbon. W. SWIENTOSLAWSKI and B. ROGA (Przemysl Chem., 1928, 12, 18—31).—

An apparatus for the determination of the ignition point of coke, coal, graphite, wood charcoal, etc. is described. The ignition point is defined as being that temperature at which the given combustible substance begins to react vigorously with pure oxygen, and is shown to have a constant value for each individual substance. R. TRUSZKOWSKI.

Ignition point of wood charcoals. W. SWIENTOSLAWSKI and M. CHORAŻY (Przemysl Chem., 1928, 12, 31—37).—The ignition point of birch charcoal rises proportionately to the temperature of carbonisation previously employed. If a sample of charcoal be heated at a higher temperature than that at which it was carbonised, the new ignition point is identical with that which would have been obtained had the wood been carbonised at the higher temperature. If wood charcoal be heated in presence of oxygen at a temperature close to its ignition point, the latter rises continually with duration of heating. R. TRUSZKOWSKI.

Ignition points of active charcoals. W. SWIENTOSLAWSKI and B. ROGA (Przemysl Chem., 1928, 12, 38—39).—The ignition points of active charcoals bear no relation to their adsorptive power, but are strictly dependent on the temperature of activation. Thus charcoals activated at 450° and at 1000° have ignition points of 169° and 457° respectively. R. TRUSZKOWSKI.

Thermal disintegration of gaseous hydrocarbons. S. MANTEL (Przemysl Chem., 1928, 12, 333—342).—The hydrocarbons contained in illuminating gas and natural gas are resolved into their elements at 1600°. The resulting gas consists of hydrogen contaminated with 0.7% of methane. R. TRUSZKOWSKI.

Occurrence and detection of hydrogen in mine air. HEYER (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 78—79).—Hydrogen is frequently a constituent of mine atmospheres, although seldom in amount sufficient to cause accidents. Small amounts of it may be detected by the fact that it reduces sodium molybdate in weakly acid solution with formation of a blue coloration. To prepare the reagent 1 g. of molybdic anhydride is dissolved in caustic soda solution, 0.1 g. of colloidal palladium mixed with sodium protalbinat is added, the solution is diluted to 200 c.c., and acidified with dilute sulphuric acid till the palladium coagulates. The test may be rendered quantitative by comparison with gases of known hydrogen content. S. BINNING.

Wood-gas-producer tar. I. J. POSTOVSKI, N. A. APOLLOV, and B. P. LUGOVKIN (Ann. Inst. Polyt. Ural, 1928, 6, 241—244).—A sample of tar from a gas producer using various species of pine wood as fuel showed a mean moisture content of 50%, 14.72% of neutral products, 0.1% of bases, and 31.8% of phenols. The components separated were *p*-cresol, *p*-ethylphenol, a dimethoxy-*p*-xylene (?), an unidentified phenolic compound, an unsaturated hydrocarbon (C₈H₆)_n (? naphthylfulvene), and 40% of resinified products insoluble in ether. The formation of the last, and the absence from the tar of the products usually found in wood tar, are ascribed to the high temperature prevailing in the producer. T. H. POPE.

Kraemer-Sarnow method [for determining the softening point of pitch] and mercury poisoning. H. MALLISON (*Z. angew. Chem.*, 1928, **41**, 839–840).—The danger of chronic poisoning by prolonged contact with mercury has been pointed out by Stock and others; it is therefore suggested that the mercury method of determining the softening point of pitch should be universally replaced by the American "ring and ball" method.

A. R. POWELL.

Crude oils from the district of the Kruimskaya settlement (Kuban, Black Sea district). A. O. YURKOV (*Neft. Choz.*, 1928, **14**, 217–219).—The wells are rich in gas, but the portion having b.p. $< 100^\circ$ is low; the kerosene fraction is satisfactory in yield but has high *d*. Kruimskaya oil contains less aromatic substances than Maikop crude oil.

CHEMICAL ABSTRACTS.

Lubricating oils from Surakhani crude oil. A. N. SACHANOV (*Azerbeid. Neft. Choz.*, 1928, No. 1, 50–52).—The light fractions consist of naphthenes and the heavy of paraffins; the fraction (mazout) available for lubricating oil is 54.2%. On distillation in a vacuum 72% was recovered as oil. Data for the cylinder stock obtained from the residue, and for the above fractions, are recorded. CHEMICAL ABSTRACTS.

Pyro-benzine from mazout from Surakhani paraffin oils and from residue from heavy gas tars. N. NOVIKOV-VAKULENKO (*Azerbeid. Neft. Choz.*, 1928, No. 1, 55–59).—Atomised heavy residues, blown against heated fire-brick, left at 617 – 774° ; 33.5–54% of gas was produced, and the residual tar was distilled. Fractional yields and densities are recorded for solar oil, mazouts, and heavy gas tar. The gases from solar oil and paraffin mazout contained, respectively, CO_2 0.7–1.3, 0.8; CO 0.7–0.5, 1.0; O_2 1.1–1.6, 1.8; H_2 9.2–25.4, 16.8; CH_4 46.7–36.5, 36.1; heavy hydrocarbons, 38.8–27.6, 20.8; N_2 2.8–7.1, 22.7%.

CHEMICAL ABSTRACTS.

Cracking of mazouts from heavy crude oils. A. N. SACHANOV and M. D. TILITSCHIEV (*Neft. Choz.*, 1928, **14**, 328–353).—On cracking at 425° and 10–60 atm., the speed at which gasoline and kerosene are formed is practically the same for all mazouts and heavy crude oils. At 425° 8–10 and 18–20% of gasoline and 12–15 and 18–22% of kerosene are formed in 30 and 60 min., respectively; the rate of cracking is doubled every 10° . Various crude oils were examined. The relation between the yields of gasoline and coke depends on the oil used, and is independent of the temperature.

CHEMICAL ABSTRACTS.

Liquefaction of petroleum asphalt by Bergius' method. K. SMOLEŃSKI and W. BADZYŃSKI (*Przemysł Chem.*, 1928, **12**, 117–136).—Petroleum asphalt when heated at 400° for 12 hrs. with hydrogen at above 100 atm. yields 60% by weight of liquid hydrocarbons, similar to those obtained by the distillation of Galician light petroleum, and of which 32% boils below 150° and 36.5% from 150° to 300° . The solid residue (25–30%) contains 87% C and 4% H, and is a good substitute for coke. About 15% of the original weight of asphalt is evolved as gaseous products containing about 40% CH_4 and 40% H_2 . Somewhat lower yields of liquid

hydrocarbons are obtained by the substitution of water-gas for hydrogen in the above reaction.

R. TRUSZKOWSKI.

Extraction of high-quality cylinder oils from lubricating oil residue. B. E. VORONOV (*Neft. Choz.*, 1928, **14**, 213–216).—Lubricating oil residues are mixed with petroleum (15%) and treated with 92.5% and fuming sulphuric acid, the petroleum then being removed, the residue treated with fuller's earth, and mixed with petroleum (15%) and naphthenic acids (0.5%). After heating at 90° and removal of water, sulphuric acid is added, the mixture agitated with air, and the acid sludge removed after 2 hrs. The treatment is repeated, sodium hydroxide solution and water being finally employed. The yield, after treatment with fuller's earth, was 60% of an oil having flash point (Brenken) 305 – 310° , viscosity (Engler, 100°) 6–6.5, carbon (Konradson) 2.2–2.4%, colour dark green. (Russ. Pat. 16,151 of 1927). CHEMICAL ABSTRACTS.

Comparison of foreign and Soviet lubricating oils. G. ANDRÉEV (*Neft. Choz.*, 1928, **14**, 351–359).—The Russian spindle oils have the lower flash point. The cold test (-20°) and the resin content are favourable.

CHEMICAL ABSTRACTS.

Treatment of bottoms from the lubricating oil fraction of Emba crude oil. N. TSCHERNOSHUKOV (*Neft. Choz.*, 1927, **13**, 188).—By treatment with sulphuric acid (2 pts.) and naphthenic acids (1 pt.), 45% of oil was separated. CHEMICAL ABSTRACTS.

Acid sludge from oil fractions from Emba crude oil. S. OBYADCHIKOV (*Neft. Choz.*, 1928, **14**, 360–362).—The tar from the washed sludge which had been heated at 130 – 150° , when treated with air at 300° gave asphalts having m.p. 80° , 60° , and 30° . The yields are recorded. CHEMICAL ABSTRACTS.

Improving viscosines (cylinder stocks). L. GUKHMAN (*Azerbeid. Neft. Choz.*, 1928, No. 2, 61–63).—"Vapours" (cylinder stocks from Emba crude oils) contain 2.1–3.1% C; "viscosines" (from Balkhanui crude oil) contain 3.0–7.0% C, and are washed with steam, treatment with alkali hydroxide being then unnecessary. The production of heavy viscosines is described, the bottoms being mixed with solar oil and treated with sulphuric acid at 20 – 25° .

CHEMICAL ABSTRACTS.

Distillation of oil shales from the liquid phase. E. VON PEZOLD (*Chem.-Ztg.*, 1928, **52**, 541–542, 562–564).—If oil shales are distilled with certain high-boiling solvents, e.g., middle or heavy shale oils boiling at 200 – 300° , the yield of light oils of b.p. below 200° is considerably greater than that obtained by heating the shale and oil separately either at 400° or under pressure. At the same time there is a decrease in the total oil distillate and an increase in the amount of coke and water formed. By continued distillation of the higher-boiling oil with fresh quantities of shale the yield of light oil first rises to a maximum and then falls to an approximately constant figure. No increase in the yield of light oil is obtained by passing the shale oil vapour over shale coke.

F. R. ENXOS.

Gasoline stripping plant. V. DENISEVICH (*Azerbeid. Neft. Choz.*, 1928, No. 1, 59–64).—The plant

is described and figured; the heat recovery is 75% and the fuel consumption 0.7%.

CHEMICAL ABSTRACTS.

Tube still for distillation of crude oil. I. N. ACKERMAN (*Neft. Choz.*, 1928, 14, 193—212).—Calculations for the construction of tube stills are given.

CHEMICAL ABSTRACTS.

Rapid corrosion test for gasoline. H. P. RUE (*U.S. Bureau Mines Rep. Invest.*, 1928, No. 2862, 5 pp.).—The sample (100 c.c.) is shaken in a 4 oz. bottle for 2 min. with 1 c.c. of mercury. The gasoline is decanted and immediately filtered, the paper dried, and the intensity of the black colour indicates the corrosiveness of the gasoline.

CHEMICAL ABSTRACTS.

"Formolite" reaction for testing mineral oils. M. BORODULIN (*Neft. Choz.*, 1928, 14, 363—364).—The precipitate dissolves when the ammonium sulphate is washed out; the use of methylal is recommended.

CHEMICAL ABSTRACTS.

Acid asphalts. S. GASIOROWSKI (*Przemysl Chem.*, 1928, 12, 90—99).—Acid asphalts, obtained as by-products of the refining of machine oils from Galician petroleum with sulphuric acid, when extracted with water to remove free sulphuric acid, yield a very stable emulsion of water in asphalt which loses water at 110°, giving a sticky, plastic mass with a mineral ash content of 0.1%. This product has acid value 8.7, saponif. value 16.6, and contains 2% of free acids, 9.6% of asphaltenes, 15% of resins, and 65% of unchanged oil. The water-soluble products of hydrolysis consist of sulphonic and asphaltogenic acids. When acid asphalt is heated at 220—230° and subjected to a current of air it becomes progressively harder and more brittle, the asphaltene content rises from 9.6 to 30.3%, the mean mol. wt. from 476 to 790, and the free oil content falls to 45.8%, the addition of free sulphur intensifying this process. The reaction of asphaltisation of mineral oils appears to be one of polymerisation associated with oxidation and formation of asphaltenes.

R. TRUSZKOWSKI.

Influence of lead tetraethyl on the combustion of mixtures of air and hydrocarbons. R. DUCHÊNE (*Compt. rend.*, 1928, 187, 200—201).—The effect of 5% of lead tetraethyl on the explosion of mixtures of hexane and air has been studied at pressures sufficiently low to prevent condensation by adiabatic compression. It is concluded that the action of the lead tetraethyl occurs in the gaseous phase, and not on the surface of drops of condensed liquid.

C. W. GIBBY.

Stills. VUISHETRAVSKI. **Rectification columns.** KOSTRIN.—See I. **Argan wood.** CABRAL.—See V. **Sulphur from gas.** CUNDALL.—See VII. **Ultramarine.** KLIMOV and LANIN.—See XIII.

PATENTS.

Treatment of carbonaceous and other materials. F. FRANK, Assr. to W. W. KEMP (U.S.P. 1,677,757—8, 17.7.28. Appl., [A] 1.12.20, [B] 2.1.23).—(A) Chemical reaction between one constituent of a heat-dissociable carbonaceous material and another present in excess is effected by the direct action of a flame produced by burning a previously formed mixture of uniform com-

position in an air-tight chamber. (B) The flame is produced by burning a uniform mixture of combustible material, a supporter of combustion, and an excess of a reducing agent in an air-tight chamber.

H. S. GARLICK.

Reducing materials by heat. Producing combustible gas. W. W. KEMP (U.S.P. 1,677,784—5, 17.7.28. Appl., 7.11.21).—(A) Carbonaceous material is passed through a heated retort, from which air is excluded, in which it comes into direct contact with a flame from the burning of a uniform mixture of a combustible material with a minimum quantity of a supporter of combustion. (B) Gas-producing material is charged into a gas generator from which air is excluded, and heated by a flame produced by the combustion of premixed gas and air in which the proportion of oxygen is sufficient only to effect combustion of the gas mixture.

H. S. GARLICK.

Treatment of coal, peat, lignite, shale, etc. with alkaline solvents. T. D. KELLY (B.P. 293,578, 29.7.27).—The material is treated with a hot alkaline solution of sodium, potassium, or ammonium oleate and then with aluminium sulphate. The solids are separated from the liquid and, suitably prepared, can be used as fuel, whilst from the liquid can be isolated fuel oil or dyes, or it can be used as a waterproof dressing.

H. S. GARLICK.

Binding materials for agglomerating finely-divided fuels or other pulverulent matter. Briquetting finely-divided coal, fuel, or other pulverulent materials. O. REYNARD and F. F. TAPPING (B.P. 293,135—6, 4.4.27).—(A) A binding agent is made by neutralising concentrated sulphite-cellulose waste liquor with alkali, adding carbonaceous material to form a paste or, alternatively, emulsifying with a petroleum or tar oil, and incorporating the product with molten bituminous material at a temperature above 100°, and under pressure. (B) The finely-divided material is mixed with 3—6% of a bituminous binder, preferably one melting at or above 120°, and with 1—4% of a water-in-oil emulsion. The latter may contain 1 pt. of fuel oil and 1—6 pts. of an aqueous solution of a suitable emulsifying agent, e.g., soda ash, sodium caseinate, sulphite-cellulose waste liquor, etc.

A. B. MANNING.

Apparatus for carbonising pulverised fuel. J. N. BAILEY, and METROPOLITAN-VICKERS ELECTRICAL CO., LTD. (B.P. 293,106, 29.3.27).—The powdered fuel is introduced into a retort of inverted pear shape, having a ring of tangential gas inlets around the wall at the part of greatest diameter. The fuel is supplied from a hopper at the top of the retort to a central distributing nozzle from which it is sprayed upwards and tangentially by the aid of compressed gas. Hot gases injected through the gas inlets whirl the fuel in a vortex within the retort and at the same time carbonise it. The coked fuel and the volatile products escape through an axial outlet at the bottom of the retort, and pass to a separator.

A. B. MANNING.

Continuous carbonising furnace. W. M. HEBURN, Assr. to SURFACE COMBUSTION CO. (U.S.P. 1,677,136, 17.7.28. Appl., 15.2.26).—The gases from the combustion chamber pass through ports to flues surrounding

the permeable walls of an open-top heating chamber, placed above the combustion chamber. Hot gases are also passed around the muffle, the floor and walls of which are continuous with those of the heating chamber. Spent gases leave the furnace at a point remote from the muffle.

F. G. CLARKE.

Gas producers. K. A. and E. H. WIDEGREN (B.P. 280,912, 14.11.27. Swed., 22.11.26).—A down-draught gas producer, comprising an upper store chamber for the fuel and a lower, preferably rectangular, combustion chamber, has all the air inlets arranged at the same horizontal level along the upper part of each of the longer sides of the combustion chamber. The distance between these two opposing groups of air inlets is small enough to ensure a practically uniform reaction across the chamber. The fire-box may be divided into two or more such chambers, having a common store chamber and a common ash-pit. The store chamber may be made wider than the combustion chamber, so that the sides of the former pass down by and provide double walls for the latter.

A. B. MANNING.

Production of gases. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 293,413, 2.4.27. Addn. to B.P. 214,544; B., 1924, 549).—Gas is generated in a producer having a grate provided with a number of small holes through which a stream of air or steam is blown at such a rate that it prevents the finely-divided solid fuel used from falling through. The gasifying agent may in some cases be admitted through the centre of the grate only, so that the fuel bed assumes a conical form and fresh material is fed continuously to the reaction zone.

R. H. GRIFFITH.

Treatment of gases for removal of hydrogen sulphide and carbon dioxide. M. AURIG and G. BRÜCKLMAYR (B.P. 293,592, 24.8.27).—Gases are treated with an alkaline lye in a cyclic system in which the used lye is regenerated continuously by treatment with an iron compound and with lime.

H. S. GARLICK.

Gas purification. F. W. SPERR, JUN., ASSR. to KOPPERS Co. (U.S.P. 1,677,304, 17.7.28. Appl., 31.12.26).—Liquids which have been used for freeing gases from acidic impurities are exposed to the action of air in the presence of hemicellulose.

H. ROYAL-DAWSON.

Apparatus for detecting, indicating, and recording the presence of inflammable vapours or gases. H. T. RINGROSE (B.P. 293,125, 1.4.27).—A vessel having porous walls, which are, however, capable of maintaining a gas pressure for a substantial time, is so arranged that the gas under test can pass therein by diffusion. Any inflammable constituents of the gas are burnt within the vessel by means of a heated platinum filament, and the diminution in pressure so produced operates a manometer and gives a measure of the percentage of inflammable gases present. In a second form of apparatus the change in pressure, moving a collapsible diaphragm, completes a circuit which lights a lamp when the percentage of inflammable gas reaches a predetermined value.

A. B. MANNING.

Tar and a process of obtaining the same. URBANA COKE CORP. (B.P. 277,955, 8.9.27. U.S., 21.9.26).

—Between 650° and 850° carbonisation of coal produces a tar which is different from any low-temperature or gas-works tar, and is rich in valuable tar acids. A plant is described in which the coal is first heated to a temperature just below that at which it becomes viscous; it is then carried rapidly through the pasty stage to the final temperature, being treated in a vertical metal retort. The condition of the coal produced in this way enables tar of specific characteristics to be formed.

R. H. GRIFFITH.

Purification of benzol fractions. C. STILL (B.P. 288,591, 31.8.27. Ger., 13.4.27).—The deleterious and difficultly removable sulphur compounds present in benzol are stated to be formed by oxidation of the hydrogen sulphide present in the freshly produced material. The removal of this impurity from the crude benzol is effected immediately after production by a continuous process involving an aqueous alkali wash.

C. O. HARVEY.

Conversion of hydrocarbons of high b.p. into compounds of lower b.p. I. G. FARBENIND. A.-G. (B.P. 268,796, 31.3.27. Ger., 1.4.26).—In the production of low-boiling hydrocarbons from tars, mineral oils, or bitumens, the losses due to carbon deposition are largely avoided by using only cobalt, chromium, nickel, manganese, tungsten, or molybdenum in construction of the apparatus. These metals exert a catalytic effect on the reaction, and may be employed further in the form of internal heating units; additional catalysts may be distributed throughout the oil or be used as granules. The raw material is treated in the liquid phase.

R. H. GRIFFITH.

Cracking oils. C. L. PARMELEE and E. W. ISOM, ASSRS. to SINCLAIR REFINING Co. (U.S.P. 1,675,575, 3.7.28. Appl., 13.8.23).—The ordinary tubular-still cracking process is modified by introducing a heated solvent oil into the partially cracked oil at one or more intermediate stages during its passage through the coil.

C. O. HARVEY.

Cracking oils. G. L. PRICHARD, ASSR. to GULF REFINING Co. (U.S.P. 1,676,207, 3.7.28. Appl., 22.6.15).—The oil is cracked under pressure in a vessel provided with a mechanical stirring device, the drive of which passes through a packing box enclosed in a chamber through which a liquid is circulated.

C. O. HARVEY.

Cracking of hydrocarbon oils. E. C. HERTHEL and T. DE C. TIFFT, ASSRS. to SINCLAIR REFINING Co. (U.S.P. 1,677,772—6, 17.7.28. Appl., [A—C] 22.6.26, [D] 12.8.27, [E] 27.10.27).—(A) The oil is cracked under pressure by passing it through a heating zone and a primary vaporising chamber, the residue then passing to an externally unheated secondary vaporising chamber, wherein the pressure is less than in the still and first chamber. The vapours from this last chamber are condensed and recirculated through the heating zone before mixing with a fresh supply of the oil to be cracked. (B) The vapours are refluxed in contact with a down-flowing stream of untreated oil. (C) The fractionation of the vapours from the secondary vaporisation chamber is controlled by supplying suitable fractions thereto. (D) A process similar in principle to (A). (E) The reflux

condensate from the primary vaporising chamber is passed through an independent pressure-cracking plant.

C. O. HARVEY.

Manufacture of hydrocarbons, especially liquid hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 293,185, 9.6.27).—Oxides of carbon are reduced by hydrogen, or gases rich in hydrogen, in the presence of contact masses containing, in addition to metallic copper, silver, gold, or zinc, elements of the 8th periodic group, and, if desired, small quantities of other elements or compounds. The process may be carried out under ordinary or higher pressures, and at elevated temperatures, in the absence of carriers of low heat conductivity.

A. B. MANNING.

Production of liquid hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 269,521, 4.4.27. Ger., 14.4.26).—Hydrocarbons are produced from mixtures of carbon monoxide and hydrogen under pressures of 50 atm. or less in the presence of catalysts consisting of metallic sulphides. The gases are not allowed to come into contact with surfaces containing iron, but the presence of sulphur compounds does not impair the activity of the catalyst.

R. II. GRIFFITH.

Manufacture of liquid products by destructive hydrogenation of coal, tars, mineral oils, etc. I. G. FARBENIND. A.-G. (B.P. 273,712, 27.6.27. Ger., 29.6.26).—High yields of low-boiling hydrocarbons are obtained from coal, tars, etc. by consecutive catalytic cracking and hydrogenation in the solid, liquid, or vapour phase. The cracking catalyst consists of elements of groups 3 and 4 of the periodic system, copper-iron mixtures or compounds, active charcoal or silica, and other substances. The hydrogenating catalysts are cobalt, molybdenum, tungsten, and their compounds. The process consists in treatment with hydrogen and the catalyst under pressure at 300–700°, followed by the use of a different catalyst under similar conditions; it may be carried out with a large excess of hydrogen, so that the product has only a low partial pressure.

R. H. GRIFFITH.

Coking hydrocarbon oils. H. WADE. From STANDARD OIL Co. (B.P. 293,231, 9.9.27).—The oil is supplied in a thin layer to a surface made of a refractory alloy containing nickel or chromium, e.g., monel metal, iron-chromium alloys, etc., and heated to a coking temperature, e.g., 500°. The coke residue is removed mechanically by suitable scraping means. An apparatus for carrying out the process continuously is described.

A. B. MANNING.

Treatment of petroleum. W. W. GARY, Assr. to C. O. MIDDLETON (U.S.P. 1,677,440, 17.7.28. Appl., 7.6.26).—Dry, gaseous sulphuric anhydride is passed through the petroleum.

C. O. HARVEY.

Refining petroleum fractions. F. C. AXTELL, Assr. to AXTELL RESEARCH LABORATORIES, INC. (U.S.P. 1,677,425, 17.7.28. Appl., 24.9.25).—After washings with acid and alkali, the fraction is agitated with a solution containing Caro's acid, and then washed successively with concentrated sulphuric acid, water, alkali, and again with water.

F. G. CLARKE.

Oil still. E. W. ISOM and A. C. VOBACH, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,676,204, 3.7.28. Appl.,

12.2.27).—The oil is circulated under pressure from a cylindrical container through a tubular still heated by flue gases from a furnace, and thence back to the container. The tubes of the still are provided with stationary solid cores, formed from twisted rods, which thus provide a continuous annular oil passage through the tubes.

C. O. HARVEY.

Process of treating methane. G. OLIVIER, Assr. to Soc. ANON. LE PÉTROLE SYNTHÉTIQUE (U.S.P. 1,677,363, 17.7.28. Appl., 3.6.26. Fr., 3.2.26).—Methane is converted into ethylene and ethylenic compounds by heating in a thin stream to 950° max., subjecting to a vacuum, and suddenly cooling to atmospheric temperature.

H. S. GARLICK.

Production of gaseous hydrocarbons from gas mixtures containing hydrogen and oxides of carbon. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 293,572, 13.7.27).—Gaseous olefines and paraffins are obtained by interaction of hydrogen and oxides of carbon in the presence of catalysts at elevated pressures and temperatures. The catalyst consists of iron or cobalt together with a noble metal or with a difficultly-reducible oxide of a metal, particularly one of the 6th periodic group. The gas mixture is circulated at a substantially lower velocity than that required for the production of liquid hydrocarbons.

R. H. GRIFFITH.

Polymerisation of ethylene, propylene, and butylene. F. HOFMANN and M. OTTO (B.P. 293,487, 8.4.27).—Ethylene, propylene, and butylene, either as individuals, mixtures, or in solution, are treated under pressure with boron fluoride or its double compounds or solutions, and in some cases with the addition of finely-divided metals. Good yields of liquid hydrocarbons are produced.

R. H. GRIFFITH.

Pulverised fuel furnaces. WITKOWITZER BERGBAU- u. EISENHÜTTEN-GEWERKSCHAFT, and C. SALAT (B.P. 285,042, 9.2.28. Ger., 9.2.27).

[Apparatus for] burning pulverised solid fuel or atomised liquid fuel. F. L. DUFFIELD (B.P. 293,330, 31.1.27).

Transportation of coke from vertical chamber ovens or from vertical retorts. C. M. CROFT and R. H. BUXTON (B.P. 293,916, 21.4.27).

Apparatus for carbonisation. C. B. WINZER (U.S.P. 1,678,687, 31.7.28. Appl., 21.12.25. U.K., 14.1.25).—See B.P. 250,661; B., 1926, 523.

Recovering light hydrocarbons from carbonaceous material. M. J. TRUMBLE (U.S.P. 1,676,675, 10.7.28. Appl., 31.7.22).—See B.P. 221,052; B., 1924, 1004.

Refining oils and waxes. W. A. PATRICK and E. B. MILLER, Assrs. to SILICA GEL CORP. (U.S.P. 1,678,298, 24.7.28. Appl., 16.3.22).—See B.P. 195,055; B., 1924, 1005.

Apparatus for continuous distillation etc. (B.P. 293,147).—See I.

III.—ORGANIC INTERMEDIATES.

Manufacture of vanillin. A. WAGNER (Chem.-Ztg., 1928, 52, 525–526, 542–543).—A mixture of oil of cloves and potassium hydroxide solution is raised to

b.p. with constant stirring, the heating being continued for a further 37 min. with careful regulation of the temperature to a maximum of 232°. After cooling, the mass is dissolved in water, extracted with benzene after careful acidification, and the benzene recovered. The isoeugenol so obtained is acetylated and afterwards oxidised with sodium dichromate, sulphanilic acid, and sulphuric acid at 67–68°, and extracted with benzene. The extracts from several operations, after removal of 80% of the benzene, are treated with the calculated amount of sodium bisulphite solution at 22°; the bisulphite compound is separated, decomposed with sulphuric acid, and the impure vanillin is centrifuged, distilled *in vacuo*, and crystallised from water containing carbon dioxide. Suitable apparatus is described.

F. R. ENNOS.

p-Cymene. WHEELER and BOST.—See IV.

PATENTS.

Preparation of polymerised vinyl alcohol and its derivatives. W. O. HERMANN and W. HAEHNEL, ASSRS. to CONSORT. F. ELEKTROCHEM. IND. (U.S.P. 1,672,156, 5.6.28. Appl., 17.7.25).—A polymerised vinyl ester (acetate, propionate) is hydrolysed with alcoholic alkali to form a polymeride of vinyl alcohol, from which various polymeric esters may be obtained by the usual methods; the butyrate is a rubber-like product.

C. HOLLINS.

Production of hexamethylenetetramine. SCHIEFERWERKE AUSDAUER A.-G. (B.P. 286,730, 9.3.28. Ger., 10.3.27).—A solution of ammonium chloride and sodium hydrogen carbonate in contact with the latter substance in solid form (Solvay liquors are suitable) is treated with formaldehyde solution.

C. HOLLINS.

Manufacture of carbazole-2-carboxylic acid. H. GRIMMEL, ASSR. to I. G. FARBENIND. A.-G. (G.P. 442,609, 9.10.24).—Carbazole and potassium hydroxide are stirred in an iron vessel first at 250°, then in a stream of dry carbon dioxide at 285–300°. The products are carbazole-2-carboxylic acid, m.p. 320° (ethyl ester, m.p. 184°), and a little of the more soluble 4-carboxylic acid. Below 270° the latter is the main product; above 300° a dicarboxylic acid is formed.

C. HOLLINS.

Manufacture of hydroaromatic dicarboxylic acids. J. D. RIEDEL A.-G. (B.P. 273,244, 28.2.27. Ger., 22.6.26. Addn. to B.P. 265,959; B., 1928, 117).—The process of the prior patent is extended to the oxidation of alicyclic alcohols and ketones, such as tetrahydro- and decahydro-naphthols, hydrindone, etc., with production of hydroaromatic dicarboxylic acids, e.g., trans-cyclohexylene-1:2-diacetic acid, m.p. 151°, the cis-isomeride, m.p. 164°, and β -2-carboxycyclohexylpropionic acid, from decahydro- β -naphthol.

C. HOLLINS.

Manufacture of sulphonic acids [wetting-out agents]. R. SAJITZ and E. POTT (CHEM. FABR. POTT & Co.) (B.P. 263,873, 31.12.26).—Wetting-out agents are obtained by condensing naphthalene or sulphonated naphthalene with aliphatic or cyclic olefines, sulphonic groups being introduced if not already present. Naphthalene- α - and - β -sulphonic acids are heated, e.g., in concentrated sulphuric acid containing propylene and higher olefines below 100°; or naphthalene is sul-

phonated and the crude reaction product is treated at 85–90° with a mixture of tetrahydrobenzene and sulphuric acid.

C. HOLLINS.

Preparation of β -hydroxyethylnaphthylamines and their substitution products. I. G. FARBENIND. A.-G., Assees. of W. DUISBERG, W. HENTRICH, and L. ZEH (G.P. 442,310, 18.11.24).—Naphthols or naphthylamines are boiled with β -aminoethyl alcohol and aqueous bisulphite as in the usual Bucherer reaction. The following products are described: β -hydroxyethyl- β -naphthylamine, m.p. 51° (and hydrochloride); 2- β -hydroxyethylnaphthylamine-7-sulphonic acid; *N*- β -hydroxyethyl- γ -acid; 1- β -hydroxyethylnaphthylamine-4-sulphonic acid; *N*-methyl-*N*- β -hydroxyethyl-2-naphthylamine-6-sulphonic acid; and 2- β -hydroxyethylamino-8-methoxynaphthalene-6-sulphonic acid (and nitroso-compound).

C. HOLLINS.

Manufacture of condensation products [naphth-indanones] from aromatic hydrocarbons or derivatives thereof. I. G. FARBENIND. A.-G. (B.P. 267,940, 16.3.27. Ger., 16.3.26).—Aromatic hydrocarbons (particularly α -methyl-naphthalene) are condensed with a β -halogenated aliphatic acid halide in presence of aluminium chloride, and the products are cyclised by first removing hydrogen halide to give unsaturated ketones and then treating them with aluminium chloride or sulphuric acid. The process may be carried out in one or several stages. 1-Methyl-4-naphthyl β -chloroethyl ketone, m.p. 60°, from α -methyl-naphthalene and β -chloropropionyl chloride, is converted by concentrated sulphuric acid at 100° into 5-methyl- α -naphthindan-9-one. Distillation of 1-methyl-4-naphthyl- β -chloropropyl ketone, m.p. 44–45°, yields the corresponding propenyl ketone, b.p. 204–206°/12 mm., which is similarly cyclised to 5:7-dimethyl- α -naphthindan-9-one, m.p. 67–68°, also obtainable by the action of sulphuric acid on the chloroketone.

C. HOLLINS.

Manufacture of 1-anthraquinonyl ketones. I. G. FARBENIND. A.-G. (B.P. 271,884, 25.5.27. Ger., 25.5.26, and Addn. 272,225, 2.6.27. Ger., 7.6.26).—(A) A 3-aryl- or (B) a 3-arylbenzanthrone is oxidised with chromic-acetic acid. Examples are: phenyl 1-anthraquinonyl ketone, m.p. 229°, from 3-phenylbenzanthrone; 1:1'-dianthraquinonylglyoxal, R·CO·CO·R, m.p. 330–331°, from 3:3'-dibenzanthronyl; α -phenyl- β -anthraquinonylglyoxal, m.p. 181°, from 3-hydroxy-2-phenylbenzanthrone (less drastic oxidation gives in this case a substance, m.p. 237°) or from phenyl 3-benzanthronyl ketone (3-benzoylbenzanthrone).

C. HOLLINS.

Preparation of *N*-nitrosoamines of the anthraquinone series. I. G. FARBENIND. A.-G., Assees. of A. JOB and H. TESCHE (G.P. 442,312, 25.10.24).—The *N*-nitroso-derivatives of 1- and 2-methylamino-, 4-bromo-1-methylamino-, 1-*p*-toluidino-, 1- and 2-benzylamino-, and 2-anilino-anthraquinones are obtained by action of sodium nitrite or amyl nitrite on the appropriate amines in acetic acid solution. (Cf. G.P. 443,126, following.)

C. HOLLINS.

Preparation of nitro-compounds of arylamino-anthraquinones, their derivatives and substitution products. I. G. FARBENIND. A.-G., Assees. of

H. TESCHE and A. JOB (G.P. 443,126, 7.11.24).—The *N*-nitroso-derivatives of arylaminoanthraquinones (preceding abstract) are converted into nitro-compounds when heated in acetic acid, oxidation and migration of the nitroso-group to the aryl nucleus taking place. The same result is obtained by nitrosating the arylaminoanthraquinones in boiling acetic acid. Nitroarylaminanthraquinones are obtained by these methods from 1- and 2-anilino-, 1- and 2-*p*-toluidino-, 1:4- and 1:5-di-*p*-toluidino-, and 1-*p*-toluidino-4-hydroxyanthraquinones, and from 4-*p*-toluidino-*N*-methylpyridoneanthrone.

C. HOLLINS.

Obtaining absolute alcohol. D. B. KEYES, Assr. to U.S. INDUSTRIAL ALCOHOL Co. (U.S.P. 1,676,735, 10.7.28. Appl., 27.12.22).—See B.P. 206,747; B., 1924, 193.

Process of making acetic acid. H. W. MATHESON, Assr. to CANADIAN ELECTRO PRODUCTS Co., LTD. (U.S.P. 1,676,454, 10.7.28. Appl., 31.3.21).—See B.P. 132,558; B., 1919, 846.

Quinoline derivatives (G.P. 440,008). **Acridine compounds** (B.P. 293,617).—See XX.

IV.—DYESTUFFS.

***p*-Cymene.** X. 2:5-Diamino-*p*-cymene and certain new dyes. A. S. WHEELER and R. W. BOST (J. Amer. Chem. Soc., 1928, 50, 2000—2005).—2:5-Diamino-*p*-cymene hydrochloride (cf. Liebermann and Ilinsky, A., 1886, 239) is most conveniently prepared by reducing 5-*p*-sulphobenzeneazocarvacrylamine hydrochloride (A., 1928, 55) with stannous chloride and hydrochloric acid. An apparatus is described suitable for the isolation of the pale yellow free amine, which is rapidly oxidised by air and gives *p*-cymoquinone with cold ferric chloride. It yields a dihydrobromide, m.p. 330° (decomp.); dinitrate, m.p. 196° (decomp.); monochloroacetate, m.p. 144°; bisdichloroacetate, m.p. 165°; bistrichloroacetate, m.p. 171°; monobromoacetate, m.p. 148° (decomp.); dibenzene-sulphonate, decomp.; monobenzoate, m.p. 145°; mono-*o*-chlorobenzoate, m.p. 161°; mono-3:5-dinitrobenzoate, m.p. 199° (decomp.); dipicrate, m.p. 207° (decomp.); and dibenzoyl, m.p. 280°, dihiocarbamyl, m.p. 235—237°, and dicarbamyl, m.p. above 350°, derivatives. With anhydrous oxalic acid in ether it forms *p*-aminocymyl-oxamic acid, m.p. 210—211°. Disazo dyestuffs of the following colours and m.p. have been prepared by tetrazotising the amine at 0° and coupling with 2 mols. of the component named: resorcinol, brown, m.p. 168—170°; β-naphthol, violet-purple, m.p. 295—298° (decomp.); sulphanilic acid, orange-yellow, m.p. 100° (decomp.); 2-naphthol-3:6-disulphonic acid, blue, m.p. above 340°; naphthionic acid, red, m.p. 270—272° (decomp.); β-thionaphthol, buff, m.p. 138—140°. When boiled with *p*-nitrosodimethylaniline or *p*-nitrosophenol in dilute alcohol, it yields the Eurhodine (aminophenazine) dyes, C₁₈H₂₂N₄, purplish-black, m.p. 300° (decomp.), and C₁₆H₁₇ON₃, brown, m.p. 200—206° (decomp.), respectively. The colours given by the above dyes on silk, wool, and cotton are tabulated.

II. E. F. NOTTON.

PATENTS.

Manufacture of dyes [preservation in finely-divided condition]. I. G. FARBENIND. A.-G. (B.P. 269,918, 22.4.27. Ger., 22.4.26).—The finely-divided dye is mixed with up to 10% of a cellulose alkyl (methyl) ether and then retains indefinitely its fine division. The mixture may be used as paste or may be dried without coagulation or crystallisation.

C. HOLLINS.

Manufacture of benzantrones. I. G. FARBENIND. A.-G. (B.P. 268,830, 4.4.27. Ger., 3.4.26).—Anthrone or oxanthrol is condensed in sulphuric acid with ketones or esters of the general formula, R·CO·CR'·CHR'', where R and R'' may be alkyl, aryl, or alkoxy, R' may also be hydrogen, and R' may be any radical. The unsaturated ketone may be generated *in situ* from corresponding hydrates or hydrogen halides. The preparation of the following benzantrones is described: 3-methyl- (from vinyl methyl ketone, methyl β-hydroxyethyl ketone, hydroxymethyleneacetone, ethoxymethyleneacetylacetone, or ethyl ethoxymethyleneacetylacetate), m.p. 164°; 3-phenyl- (from phenyl vinyl ketone or β-chloropropiophenone), m.p. 183°; 2:3-dimethyl- (from methyl hydroxymethylene-ethyl ketone), m.p. 207°; 2-methyl-3-ethyl- (from hydroxymethylenediethyl ketone), m.p. 142°; 1:3-diphenyl- (from phenyl styryl ketone), m.p. 195—196°; 1:3-dimethyl- (from propenyl methyl ketone), m.p. 114—115°; 1-phenyl-3-methyl- (from styryl methyl ketone), m.p. 175—176°; 3-phenyl-1-methyl- (from phenyl propenyl ketone), m.p. 174—175°; 1-*p*-anisyl- (from *p*-anisyl β-chloroethyl ketone, m.p. 64°, by way of an intermediate product, m.p. 181°), m.p. 186°; 3-hydroxy-2-acetyl- (from ethyl hydroxymethyleneacetoacetate, m.p. 295°; 3-hydroxy-2-carbethoxy- (from ethyl ethoxymethylenemalonate), m.p. 206°; 3-hydroxy-2-phenyl- (from ethyl phenyl-hydroxymethyleneacetate), m.p. 230°.

C. HOLLINS.

Production of dyes [for wool and silk]. L. B. HOLLIDAY & Co., LTD., J. KITSON, and C. SHAW (B.P. 293,110, 30.3.27).—Chloranil or bromanil is hydrolysed with dilute sodium hydroxide or carbonate, the 3:6-dichloro(or dibromo)-2:5-dihydroxy-*p*-benzoquinone is filtered off, and the filtrate treated with acid or salt to precipitate a brown dye for wool or silk. Similar products are obtained by condensing chlor- or brom-anil with its hydrolysed product in alkaline solution.

C. HOLLINS.

Manufacture of chloroperylenequinones. F. BENSA (B.P. 281,281, 17.11.27. Austr., 26.11.26).—Highly chlorinated perylenes, containing 8—12 (especially 12) atoms of chlorine per molecule, are converted by 25% oleum at 150—160° into octachloroperylenequinone, an olive-green vat dye.

C. HOLLINS.

Manufacture of vat dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 292,741, 5.5.27).—Halogenated perylenetetracarboxylic dianhydrides containing less than 4 (preferably 2) halogen atoms are condensed with *o*-phenylenediamine. The vat dyes so obtained give more easily soluble leuco-compounds than the non-halogenated product (B.P. 201,786; B., 1923, 986 A) and the dyeings are faster to soap and sodium carbonate than those of the tetrachloro-compound (B.P. 248,519; B., 1926, 355).

C. HOLLINS.

Manufacture of [black] vat dyes. I. G. FARBERENIND. A.-G. (B.P. 280,846, 6.4.27. Ger., 18.11.26).—Diazobenzanthrone, prepared from the reduced nitro-compound of Example 2 of B.P. 12,518 of 1906 (cf. B., 1906, 844), is treated with potassium xanthate or other sulphurising agent, and the product is fused with alkali (alcoholic potassium hydroxide at 280°) to give an intense black vat dye. C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. J. Y. JOHNSON. From I. G. FARBERENIND. A.-G. (B.P. 290,408, 24.3.27).—2:6(or 7)-Dithiolanthraquinone is condensed with 2 mols. of 1-chloroanthraquinone-2-carboxylic acid or 1-diazoanthraquinone-2-carboxylic acid, or 2:6(or 7)-tetrazoanthraquinone is condensed with 2 mols. of 1-thiolanthraquinone, and the resulting 2:6(or 7)-bis-(2-carboxyanthraquinonylthiol)anthraquinone is cyclised to an anthraquinonebisthioxanthone by heating in concentrated sulphuric acid or boiling benzotrichloride. The products are yellow vat dyes of high tinctorial power and good fastness. C. HOLLINS.

Manufacture of orange vat dyes of the anthraquinone series. I. G. FARBERENIND. A.-G. (B.P. 280,492, 25.4.27. Ger., 15.12.26).—Orange vat dyes are produced by converting 1-acylamino-4-hydroxyanthraquinones into their ethers by treatment with hydroxyalkylating agents, e.g., *p*-toluenesulphonic esters of ethylene glycol or β -chloroethyl alcohol, glycerol, etc. From 1-benzamido-4-hydroxyanthraquinone and β -chloroethyl *p*-toluenesulphonate are obtained (a) a mixture of β -chloro- and β -hydroxy-ethyl ethers and (b) the ethylene ether of the hydroxy-compound. The ethylene ether also results when ethylene di-*p*-toluenesulphonate is used. 1-Anisamido-4-hydroxyanthraquinone gives a similar yellow-orange vat dye, which does not change shade on soaping. C. HOLLINS.

Manufacture of dyes of the anthraquinone series. J. Y. JOHNSON. From I. G. FARBERENIND. A.-G. (B.P. 292,896—7, 18.3.27).—(A) The 1:1'-dihydroxy-2:2'-dianthraquinonyl of G.P. 167,461 (cf. Scholl and others, A., 1920, i, 169) is obtained more smoothly by fusing the α -hydroxyanthraquinone with alkali in presence of an organic medium (alcohol, aniline) and with the exclusion of oxygen. Similar products are prepared from substituted α -hydroxyanthraquinones. That from 4-amino-1-hydroxyanthraquinone gives a grey, that from 1-hydroxy-4-methoxyanthraquinone a brown, on wool from a faintly alkaline vat. (B) The crude alkaline fusion products of the preceding patent may be purified by treatment with oxidising agents (hypochlorite, permanganate) and/or fractional precipitation from sulphuric acid. C. HOLLINS.

[Anthraquinone] dyestuffs and intermediates. H. W. HERWARD, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 293,328, 29.12.26).—Hydroxyanthraquinones, particularly alizarin, are produced by heating 2-methylanthraquinone or anthraquinone-2-carboxylic acid with caustic alkali at 180° in presence or absence of oxidants such as sodium chlorate. C. HOLLINS.

Manufacture of copper compounds of substantive azo dyes. W. CARPMAEL. From I. G. FARBERENIND.

A.-G. (B.P. 292,660, 18.3.27).—Copper compounds fast to alkali and ironing are obtained from azo couplings of *o*-aminophenol ethers (excluding *o*-anisidine containing a 3-hydroxyl or 3-carboxyl group). Examples of suitable azo dyes are: *o*-anisidine (2 mols.) \Rightarrow di-J-acid (reddish-violet); 4-nitro-*o*-anisidine (2 mols.) \Rightarrow di-J-acid (bluish-violet); 5-nitro-*o*-anisidine-4-sulphonic acid (2 mols.) \Rightarrow phenyl-J-acid (violet); *o*-aminophenyl benzyl ether (2 mols.) \Rightarrow carbonyl-J-acid (bordeaux); *o*-phenetidine (2 mols.) \Rightarrow di-J-acid (reddish-violet); *o*-anisidine \rightarrow carbonyl-J-acid \leftarrow aniline-*o*-sulphonic acid (red); *o*-aminophenoxyacetic acid (2 mols.) \Rightarrow di-J-acid (violet); *o*-aminophenoxyethyl alcohol (2 mols.) \Rightarrow di-J-acid (reddish-violet); aniline-2:5-disulphonic acid \rightarrow 2:5-dimethoxyaniline \rightarrow phenyl-J-acid (blue). Numerous variations of these examples are mentioned. C. HOLLINS.

Metallic compounds of *o*-hydroxyazo dyes. I. G. FARBERENIND. A.-G. (B.P. 268,754, 17.3.27. Ger., 31.3.26).—Metal compounds of *o*-hydroxyazo dyes which contain nitro- or amino-groups may be modified by reduction of nitro-groups or acylation of amino-groups without destroying the metal complex. Examples are the chromium compounds of: 4-nitro-2-aminophenol-6-sulphonic acid \rightarrow β -naphthol, reduced with sodium sulphide (bluish-grey); *p*-chloro-*o*-aminophenol \rightarrow H-acid, acetylated (reddish-blue); nitro-1:2:4-aminonaphthol-6-sulphonic acid \rightarrow β -naphthol, reduced with alkali and dextrose (dark blue); also the copper compound of picramic acid \rightarrow H-acid, acetylated (redder shade). C. HOLLINS.

Manufacture of intermediate products and vat dyestuffs derived from diaroylperylene. COMP. NAT. DE MAT. COL. ET MANUF. DE PROD. CHIM. DU NORD RÉUNIS, ÉTABL. KUHLMANN (B.P. 267,121, 1.3.27. Fr., 5.3.26).—See F.P. 612,338; B., 1927, 698.

Coloured masses etc. (B.P. 293,067).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Degumming of silk. III. Action of hydrochloric acid on the sericin and effect of formaldehyde on the action. IV. Gelatinisation of the sericin. T. TAKAHASHI (J. Soc. Chem. Ind. Japan, 1928, 31, 34—39, 39—45; cf. B., 1928, 184).—III. Raw silk was boiled for 30 min. with hydrochloric acid of various concentrations ranging from 5×10^{-3} to $95 \times 10^{-3}N$, and the amount of acid consumed and the degree of degumming (i.e., loss of weight of the fibre mass) were determined. The effect of formaldehyde on these values was also examined. The acidity of the bath, $2-3 \times 10^{-3}N$, was supposed to be the isoelectric point of sericin. The degree of degumming decreased as the acidity of the bath was increased up to this acidity, reaching a minimum at this point, and then increased; the amount of hydrochloric acid consumed, however, increased up to this point and then remained stationary even after further increase of the acidity. In the presence of formaldehyde the degree of degumming showed no minimum as the acidity of the bath increased. The curves for the degree of degumming in the presence and absence of formaldehyde, respectively, coincided at the minimum point of the latter and again further on. The amount of acid

consumed, however, in the presence of formaldehyde at first increased as the acidity of the bath increased, reached a maximum, and then decreased. It is concluded that when the acidity of the hydrochloric acid bath is less than $2-3 \times 10^{-3}N$, the sericin adsorbs hydrogen ions and becomes gelatinised; above this point it does not gelatinise, but reacts with the acid to form a soluble hydrochloride.

IV. The viscosity of the silk boiling-off liquor produced from the degumming baths of various p_H values in presence or absence of formaldehyde was examined. As the p_H value of the bath increased, the viscosity of the liquor increased and reached a maximum at a value corresponding to $3 \times 10^{-4}N$ of sodium hydroxide, and then decreased. This point agreed with the maximum point of gelatinisation. In the presence of formaldehyde, liquors of low viscosity were obtained indicating the destruction thereby of the gelatinising properties of sericin. It is concluded that degumming is mainly due to gelatinisation of sericin in the alkaline liquor, and to swelling in the acid liquor.

S. OKA.

Composition and use [distillation] of argan wood.

A. DA C. CABRAL (Bull. Inst. Pin, 1927, 289; Chem. Zentr., 1928, i, 1295).—The dry wood of *Argania sideroxylon* (Morocco) yields: lignin 32.36, α -cellulose 50.90, $\beta + \gamma$ -cellulose 5.10, ether extract 0.27%. Dry distillation (8 hrs. at 500°) yields carbon 28.1, anhydrous tar 4.9, soluble tar 2.0, acetic acid 2.2, methyl alcohol 0.7%. The tar affords oil 40.1, residue 51.7, gas 8%.

A. A. ELDRIDGE.

Investigation and purification of sulphate-turpentine. I. J. POSTOVSKI and V. G. PLJUSNIN (Ann. Inst. Polyt. Ural, 1927, 6, 245–258).—The turpentine oil obtained in abundance in the preparation of cellulose by the sulphate process may be readily purified from compounds which give precipitates with mercury salts (mercaptans and sulphides) by distillation in a current of steam. The first 15% of the distillate contains 86% of these sulphur compounds, and if 50% of the oil is distilled only 2% of the sulphur compounds remains. The purified sulphate-turpentine is a colourless liquid of characteristic, pleasant odour, of initial b.p. 153° , d_{15}^{20} 0.8645, n_D^{20} 1.46728, $[\alpha]_D^{20} +14.47^\circ$. It contains 90% of α - (and β -) pinene and not more than 1% of dipentene, and is suitable for the preparation of paints and varnishes, and for pharmaceutical purposes.

T. H. POPE.

Cellulose ester solvents. II. H. WOLFF [with G. ZEIDLER and W. TOELDT] (Farben-Ztg., 1928, 33, 2668–2672).—Solvents of the glycol type (cf. B., 1928, 519) were further investigated from the practical point of view. The conditions under which a tendency to irregularity appears in the films of lacquers containing these solvents and varying amounts of diluents, and the correcting of the irregularity by the addition of butyl alcohol and plasticisers are described. The use of the glycol solvents, particularly "ethyl glycol," in lacquers induces good "insulation" from oil undercoats, whilst improving the rubbing qualities and elasticity of the lacquer film. The whitening effect produced by "methyl glycol" may be counteracted by suitable solvent mixtures, or alternatively may be made use of

to obtain opacity with a smaller concentration of pigment than would otherwise be necessary. The mechanical properties of such films are in no way inferior to those of corresponding films containing butyl acetate as solvent.

S. S. WOOLF.

Detergent experiments on cotton. CHAPIN.—See XII.

PATENTS.

Obtaining textile fibres of high quality from the skins of fish, e.g., *Chondropterygii selachii*. A. EHRENREICH (B.P. 284,297, 27.1.28. U.S., 27.1.27).—The fleshed skins of fish, particularly those of the plagiotomi such as sharks, are treated for a short period with lime, then subjected to the diastatic action of enzymes such as an extract of trypsin or of shark glands for 24–30 hrs. at $35-40^\circ$ whereby the intercellular substances are disintegrated and the adhesion between the fibres of the skin is reduced; the skins are then rinsed and tanned, and the fibres separated by hand or mechanically. It is advantageous to treat the skins, immediately after their treatment with enzymes, with a colloid such as gum tragacanth or tragasol. The resulting fibres are durable, have a high lustre, and are capable of being printed, dyed, and finished.

A. J. HALL.

Saccharification of cellulose-bearing material. J. PERL, Assr. to M. M. CORY (U.S.P. 1,677,046, 17.7.28. Appl., 3.7.25).—Cellulosic material is disintegrated by treatment with a counter-current of anhydrous hydrogen chloride diluted with a cooled inert gas.

A. J. HALL.

Manufacture of coloured cellulose plastics and solutions thereof. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 293,485, 8.4.27. Addn. to B.P. 247,288; B., 1926, 315).—Large amounts of colouring substances (up to 25%) are incorporated in the plastic masses prepared by methods described in the chief patent, so that the resulting products when diluted with solvents or other substances do not require the further addition of colouring substances.

A. J. HALL.

Process of making paper. PLASTIC, INC., Assees. of A. L. KENNEDY (B.P. 287,538, 8.6.27. U.S., 25.3.27).—Paper having superior resistance to electricity, heat, cold, oils, and moisture is manufactured by incorporating alginic acid in a soluble form in a fibrous pulp, then precipitating the alginic acid by the addition of a salt (e.g., zinc sulphate or calcium acetate) or lime or a mineral acid, washing the product, and converting it into paper by the usual methods.

A. J. HALL.

Production of high- α -cellulose fibre. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,678,230, 24.7.28. Appl., 28.4.25).—See B.P. 278,767; B., 1927, 935.

Filters for the fluid material for production of artificial textile threads or fibres. H. KINDERMANN (B.P. 293,816, 12.4.27).

Spinning machines for artificial silk. C. HAMEL A.-G. (B.P. 284,986, 14.9.27. Ger., 7.2.27).

Drying apparatus for textile goods etc. E. J. ROWE (B.P. 293,954, 9.6.27).

Binding materials (B.P. 293,135–6).—See II. Tanning materials (F.P. 620,394).—See XV. Nitration of cellulose (B.P. 293,190).—See XXII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Dyeing of mixed textile goods. I. G. FARBENIND. A.-G. (B.P. 267,985, 22.3.27. Ger., 27.3.26).—Level shades on silk-wool mixtures are obtained by using sulphonated mono- or dis-azo couplings of phenyl-pyrazolones containing in either or both components one or more ether groups. Examples are: 1-*p*-sulphophenyl-3-methyl-5-pyrazolone coupled with *o*-anisidine, *o*-aminodiphenyl ether, 3-aminotolyl benzyl ether; aniline-*o*-sulphonic acid \rightarrow 1-*p*-anisyl-3-methyl-5-pyrazolone; 4-amino-2-sulphodiphenyl ether \rightarrow 1-phenyl-3-methyl-5-pyrazolone. C. HOLLINS.

Production of fast dyeings from dyes of the anthraquinone series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 292,658, 18.3.27).—Wool, cotton, and artificial fibres are dyed with 1:1'-dihydroxy-2:2'-dianthraquinonyl (from alkaline fusion of α -hydroxyanthraquinone; G.P. 167,461) from a faintly alkaline vat in fast yellow shades. The products of B.P. 292,896—7 (B., 1928, 667) may be used with advantage in this way. C. HOLLINS.

Dyeing mixed textile goods. H. WAGNER, H. EICHWEDE, and E. FISCHER, Assrs. to GRASELLI DYESTUFF CORP. (U.S.P. 1,678,611, 24.7.28. Appl., 28.1.26. Ger., 7.2.25).—See B.P. 247,224; B., 1927, 520.

Dyeing with vat and azo dyes. W. WINTERHALDER, Assr. to GRASELLI DYESTUFF CORP. (U.S.P. 1,678,580, 24.7.28. Appl., 15.5.26. Ger., 20.5.25).—See B.P. 252,384; B., 1927, 775.

Dyeing apparatus [for hat bodies etc.]. V. BÖHM (B.P. 270,758, 9.5.27. Austria, 7.5.26).

Sulphonic acids (B.P. 263,873).—See III. **Printing colours etc.** (B.P. 292,665 and 292,894).—See XIII.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Preparation of mixed acid. VON BEZOLD (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 190—193).—Methods for preparing mixed acids are described. Continuous mixing may be carried out in the turbine apparatus described by Neumann (G.P. 274,854). This apparatus was designed for continuous nitration of toluene, but it is equally suitable for acid mixing. Another type of mixer is described which has no moving parts. The mixing takes place in a cooled azidur coil into which the nitric and sulphuric acids are fed together with previously prepared mixed acid. By using two sets of measuring vessels the mixing can be made practically continuous. S. BINNING.

Catalytic oxidation of ammonia. VII. L. ANDRUSOV (Z. angew. Chem., 1928, 41, 205—206; cf. A., 1927, 1039).—A reply to criticism by Raschig of previous work (cf. B., 1927, 906). The author re-states his reasons for believing that nitroxyl, HNO, is an intermediate product of the catalytic oxidation of ammonia. W. A. RICHARDSON.

Catalytic oxidation of ammonia. F. RASCHIG (Z. angew. Chem., 1928, 41, 207).—A reply to Andrussov (see preceding abstract).

Regulating the density of milk of lime. L. DYACHUK (Nauch. Zapiski, 1928, 5, 285—287).—A new type of automatic regulator is described.

CHEMICAL ABSTRACTS.

Electrolytic removal of iron from aluminium salts. L. WASILEWSKI and S. MANTEL (Przemysl Chem., 1928, 12, 48—51).—Iron present as an impurity in aluminium salts can be separated electrolytically using a mercury cathode; in this way 3 mg. of ferric oxide per 100 g. of aluminium oxide can be separated. The iron is present in colloidal solution in the mercury cathode, from which it can be separated by filtration through chamois leather. The residue after distillation of the mercury contains 1.27% of aluminium.

R. TRUSZKOWSKI.

Removal of iron from aluminium salts by recrystallisation. S. ZABICKI (Przemysl Chem., 1928, 12, 77—90).—Ammonium alum containing only 0.002% of ferric oxide is obtained in 86% yield by three successive recrystallisations, using concentrations of not above 68% of alum; above this concentration supercooling is liable to occur. Rapid stirring favours the formation of small crystals, thereby yielding a purer product.

R. TRUSZKOWSKI.

Making pure tungstic oxide [from wolframite]. W. B. GERO and C. V. IREDELL (Chem. Met. Eng., 1928, 35, 412—417).—Wolframite concentrates containing 65—75% WO₃ are ground and digested with concentrated boiling caustic soda, calcium chloride is added to the resulting solution, and the calcium tungstate digested with boiling hydrochloric acid. The tungstic acid is converted into ammonium paratungstate and the latter ignited. The lay-out of a plant to produce tungstic oxide of high purity by this process is discussed. Combined mechanical and steam agitation is employed in the caustic soda treatment of the ore, which is very heavy. The hydrochloric acid treatment of the calcium tungstate is performed in a concrete tank with internal lining of acid-proof brick backed by asphalt. A rubber-covered agitator and steam pipe for heating are used, together with a stoneware outlet closed by a hard rubber plug for tungstic acid removal. The clear solution is siphoned off. The dissolution of tungstic acid in ammonia and the subsequent filtration are carried out in wooden vessels, and all pipe lines in this stage are of hard rubber; the precipitation of the ammonium paratungstate is carried out in a rubber-lined steel tank. The necessary hydrochloric acid is added through 24 fine jets up to neutrality, the precipitate washed by decantation, drained on wooden vacuum filters, and dried on glass trays. The ignition is carried out on fused silica trays. The finished product contains over 99.98% WO₃. C. IRWIN.

Drying sulphur recovered from manufactured gas. K. N. CUNDALL (Chem. Met. Eng., 1928, 35, 407—411).—Sulphur is now being recovered as a paste by the liquid purification process at many gas works in Western America. It is very finely divided and contains 55% of moisture. If this paste is treated with steam in a digester at about 119° the sulphur is flocculated and partial drying results. Addition of alkali is necessary to neutralise sulphuric acid formed. The partly melted

sulphur is dark in colour and contains 25% of moisture, but is suitable for the manufacture of lime-sulphur preparations. A rotary gas-fired dryer for complete drying was abandoned owing to the discovery that excessive heating detracted from the toxicity of the sulphur dust. A steam-heated tunnel dryer in which the sulphur was treated in the form of press cakes was found satisfactory. The quantity dried was 0.2 lb. per sq. ft. of tray space per hour and the loss was small. The sulphur can also be coagulated by boiling, after which it may be partially dried by centrifuging. This sulphur is much more toxic than other forms and more finely divided, most of the particles being below 15 μ .

C. IRWIN.

Iron carbonyl. MITTASCH.—See X. **Electrolysis of water.** NOEGGERATH.—See XI. **Tannery investigations.** GRASSER.—See XV.

PATENTS.

Manufacture of arsenic acid. F. ULLMANN and G. TREWENDT, Assrs. to J. MICHAEL & Co. (U.S.P. 1,677,251, 17.7.28. Appl., 26.6.23. Ger., 6.3.23).—A mixture of 10 pts. of arsenic trioxide, 20 pts. of water, and 3.8 pts. of potassium chlorate is heated to boiling, and a small amount of hydrochloric acid is added.

H. ROYAL-DAWSON.

Synthetic manufacture of ammonia. I. G. FARBENIND. A.-G. (B.P. 267,554, 12.3.27. Ger., 13.3.26).—The gases are brought into contact with a catalyst of pure iron produced by thermal decomposition of iron carbonyl, and a small percentage of alumina, magnesia, zirconium oxide, or potassium oxide as activating agent.

W. G. CAREY.

Manufacture of alkali nitrates. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 293,495, 12.4.27).—An alkali nitrite is treated with oxygen or air at 300–500° in presence of a catalyst, *e.g.*, an alkali hydroxide or an alkaline-earth oxide, intimate contact being obtained by using an easily liquefiable catalyst or by melting the nitrite; the action below 360° can be facilitated by using an eutectic mixture, *e.g.*, caustic potash and caustic soda, melting at 180°.

W. G. CAREY.

Manufacture of chromium oxide and chromium hydroxide. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 293,494, 12.4.27).—An aqueous solution of an alkali chromate is heated above 110°, under pressure, with an organic reducing agent, *e.g.*, a carbohydrate soluble in water, or sawdust, and the resulting hydroxide is washed, dried, and ignited.

W. G. CAREY.

Production of anhydrous chlorides free from oxides. A. L. MOND. From I. G. FARBENIND. A.-G. (B.P. 293,410, 6.4.27).—Oxides, substances containing oxides, or chlorides which on heating decompose into oxides are mixed with reactive carbon, made into a suspension in fused chlorides and caused to flow down a heated irrigation tower in counter-current to hydrogen chloride, chlorine, phosgene, etc. The coke filling of the tower serves as resistance for the electric heating.

W. G. CAREY.

Carrying out chemical reactions in which reaction gases are circulated. SYNTHETIC AMMONIA & NITRATES, LTD., and J. HUGHES (B.P. 293,138, 6.4.27).—The

invention is illustrated by the following example. Gas removed from a synthetic ammonia plant containing an undue accumulation (5%) of useless argon and methane is scrubbed with kerosene at the working pressure of 200 atm., the quantity of oil used being 10 cub. m. per 1000–5000 cub. m. of gas, and the temperature normal or lower. The scrubbed gas is returned to the ammonia plant. The pressure of the liquid is let down in a first stage to 3 atm., in a Pelton wheel if desired, and the gas released will contain roughly 25% each of methane and nitrogen and 40–50% of hydrogen. In the second stage to atmospheric pressure the gas evolved will contain 50–60% of argon, the rest nitrogen; this fraction may be of value as a source of argon.

B. M. VENABLES.

Process of making colloidal iodine. R. W. JAMES. From MERCK & Co. (B.P. 293,504, 20.4.27).—Iodine is dissolved in alkali hydroxide solution, gum arabic (2% of the iodine present) is added as a protective colloid, and colloidal iodine is precipitated by adding hydrochloric acid.

W. G. CAREY.

Converting hydrogen sulphide into sulphur dioxide. H. BÄHR, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,678,630, 31.7.28. Appl., 4.5.25. Ger., 6.5.24).—See F.P. 609,931; B., 1927, 252.

Process of making a mixture of nitrogen and hydrogen. F. G. LILJENROTH (U.S.P. 1,678,518, 24.7.28. Appl., 17.10.22. Swed., 3.12.21).—See B.P. 189,789; B., 1923, 1071.

Catalytic reactions (B.P. 289,759).—See I. **Rotary kiln** (B.P. 293,331).—See IX. **Phosphate fertilisers** (B.P. 282,619).—See XVI.

VIII.—GLASS; CERAMICS.

Ancient glasses. III. B. NEUMANN (Z. angew. Chem., 1928, 41, 203–204).—Analysis of glasses from the Babylonian city of Nippur, and dating from about 250 B.C., shows that the samples fall into three groups, distinguished by the colours blue, green, and rose. The chemical compositions of members of the same group are similar, but there are considerable differences between the groups. All contain the sulphur characteristic of ancient glass, and high proportions of iron and alumina. The alkali content is exceptionally low (9–13% Na₂O) and all contain copper and manganese. The rose colour is ascribed to manganese, the blue to copper and manganese, and the green to interaction between alkali sulphide (the content of which is high in these glasses) and copper or manganese oxide.

W. A. RICHARDSON.

Casing of clear by opal glass. Importance of annealing. F. WINKS and W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 161–163).—The coefficients of expansion of a clear glass and of an opal glass which had shown a tendency to crack off when used for flashing this were in close agreement over the normal range. On the other hand, the lower and upper annealing temperatures of the opal were 30° and 53° respectively above that of the colourless glass.

A. COUSEN.

Melting of glass. R. HEMINGWAY (J. Soc. Glass Tech., 1928, 12, 131–134).—In a cross-flame regenera-

tive oil-fired tank furnace, good colourless glass had been obtained at the rate of 1 ton per 9 sq. ft. of melting area per 24 hrs., with a maximum safe peak-load rate of 1 ton per 8 sq. ft. The corresponding rates for an amber glass were 6 and 5 sq. ft. respectively, and the increased melting rate in this case was found to be due to the sulphur employed in the batch. A. COUSEN.

Acceleration of glass melting by the use of volatile constituents in the glass batch mixture. W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 134—138).—The batch tested was of a simple soda-lime type, and the volatile agents added were ammonium chloride, nitrate, or sulphate. All accelerated the melting rate in pot furnaces, whilst the chloride and sulphate also accelerated fining. Results were less effective in tank furnaces.

A. COUSEN.

Design and operation of glass furnaces. W. W. WARREN (J. Soc. Glass Tech., 1928, 12, 128—131).—Means of improving the efficiency of glass-melting furnaces, as by insulation and utilisation of waste heat, the provision of measuring and regulating instruments in the furnace system, etc., are advocated. The regenerative pot furnace is more efficient than the recuperative type, and, in pot melting, the plunging of glass during founding with arsenious oxide improves the fining rate.

A. COUSEN.

Control and distribution of temperature in lehrs. A. COUSEN, H. W. HOWES, and F. WINKS (J. Soc. Glass Tech., 1928, 12, 146—158).—Essential temperature conditions within a lehr for the successful and economical annealing of glass are discussed. Temperature conditions along lehr belts have been explored by the use of a nichrome-kromore traveller thermocouple, by means of which the temperature distribution generally holding in direct-fired, semi-muffle and muffle lehrs has been found.

A. COUSEN.

Physicochemical examination of the Borovitchi refractory clays. G. G. URAZOV and N. I. VLODAVTZA (Ann. Inst. Anal. Phys. Chem., 1927, 3, 725—745).—Plastic clays have a higher water content than friable clays, as well as a higher titanium, iron, and alkaline-earth content. Three breaks may be observed in the temperature-time curves obtained by heating the clays to 1100°. The first two, indicating endothermic effects, occur at 100—110° and 400°, and are due to the loss of adsorbed and constitutional water respectively. The third, due to an exothermic reaction, takes place at 950° and is less marked for friable than for other clays. Curves connecting degree of dehydration of air-dry refractory clays with temperature show that at 400° 90% of the water originally present is given up; the remainder disappears at 520—600°. It is suggested that these clays consist of kaolinite, and that the air-dry clay ($\text{Al}_2\text{Si}_2\text{O}_7 \cdot n\text{H}_2\text{O}$) between 100° and 400° continually loses water, the resulting substance being hydrated leverrierite, $\text{Al}_2\text{Si}_2\text{O}_7 \cdot m\text{H}_2\text{O}$, which at about 600° is completely dehydrated. X-Ray measurements of the angles of the crystal lattices of various Russian clays show that these are very close to one another and to those of nacrite.

R. TRUSZKOWSKI.

Decomposition of clays. L. WASILEWSKI (Przemysl Chem., 1928, 12, 40—48).—The decomposition of clays

and kaolin by ammonium sulphate proceeds as if ammonium hydrogen sulphate and ammonium alum are first produced (up to 357°), then ammonia and ammonium sulphate are driven off from the alum (420—430° to 450°). Up to 360° about 0.5% of the ammonia is oxidised, and ammonium alum is leached out of the products, whilst at 450° 9% of ammonia is oxidised and aluminium sulphate is obtained. Clays with a high iron content release ammonia more readily, whilst those free from iron resist conversion into aluminium sulphate. CHEMICAL ABSTRACTS.

Firing of clays in the presence of water vapour and sulphur dioxide. J. KONARZEWSKI and B. KRYŃSKI (Przemysl Chem., 1928, 12, 176—184).—Below 550° the dehydration of china clay is retarded by the presence of water vapour; above this temperature the velocity of dehydration is unaffected by this factor. The presence of sulphur dioxide has no influence on the velocity of dehydration; the fired clay, however, contains in this case sulphates of calcium, magnesium, aluminium, and iron. The sulphur can be entirely eliminated by refiring in an oxidising atmosphere. Alumina and ferric oxide can to a certain extent be extracted from fired clay by hydrochloric acid.

R. TRUSZKOWSKI.

Adsorption phenomena with clays in non-aqueous media. B. NEUMANN and S. KOBER (Kolloidchem. Beih., 1928, 27, 1—43).—Measurements of the adsorption of a silver xylosol have shown that the peculiar relations of heated clay towards the adsorption of non-aqueous dye solutions and vegetable oils are to be traced to the different degrees of dispersion. A differentiation is made between the adsorption of crystalloidal and colloidal matter by clays. Investigation of the adsorption of a large number of substances showed a continuous progression from crystalloidal to colloidal behaviour. Silver xylosol and a gold-resin sol furnish examples of purely colloidal adsorption. Curves relating the adsorption of gold sol to the temperature of heating are given for some clays, and irregularities are traced to gas adsorption. It is concluded that the essential alteration taking place in clays on heating is a reversible change in capillary structure.

E. S. HEDGES.

Analysis of silicates by decomposition with alkali hydroxides in a nickel crucible. C. J. VAN NIEUWENBURG and H. H. DINGEMANS (Chem. Weekblad, 1928, 25, 266—268).—Either sodium or potassium hydroxide in the molten condition effects practically complete decomposition of most silicates at temperatures of about 400°, at which nickel is very little attacked if it be kept out of direct contact with the flame. The nickel crucible may therefore be used for silicate analyses by this method, and the fused mass boiled with water in the crucible itself.

S. I. LEVY.

Dolomite for steel works. JACOBS.—See X.

PATENTS.

Manufacture of compound glass. E. GEISEL (B.P. 293,671, 27.3.28).—The products of polymerisation of methyl or ethyl acrylate are used as a uniting layer between sheets of glass, or as a coating to a single sheet. A. COUSEN.

Clay product, ceramic composition, and associated processes. VITREFRAX Co., Assees. of T. S. CURTIS (B.P. 273,258, 16.5.27. U.S., 22.6.26).—A natural aluminium silicate, such as cyanite, the crystals of which expand in the direction of their major axes, at the temperature of firing, is mixed with the ceramic mass before moulding. The product is strengthened by the resulting network of elongated crystals, and no shrinkage takes place. To prepare the cyanite, the crystals are heated at 927–982°, subjected to table-concentration with water, and then milled. Alternatively, the red-hot crystals are quenched in cold water, whereby the bulk of the impurities are separated without breaking the crystals transversely.

F. G. CLARKE.

Up-draught process of and muffle kiln for firing. T. DOWNS (U.S.P. 1,677,818, 17.7.28. Appl., 25.1.27).—The air for drying passes upwards around the sides of the kiln and over its domed roof. The air also passes beneath the kiln and up through a central vertical flue. The various air currents can be regulated.

F. G. CLARKE.

Manufacture of splinterless, reinforced glass. H. LE V. LAWRENCE, and KENILWORTH MANUF. CO., LTD. (B.P. 293,666, 1.3.28).

IX.—BUILDING MATERIALS.

Mechanism of setting and hardening of cement. T. MAEDA (Sci. Papers Inst. Phys. Chem. Res. Tokyo, 1928, 8, Suppl., 5–7).—Four processes are involved in the setting and hardening of cement. These are the dissolution of an unstable solid phase in a liquid phase, the formation of a solution supersaturated with regard to the stable solid phase, the separation of a new stable solid phase, in particles of colloidal dimensions, which may adsorb certain constituents of the liquid phase, and the dissolution of further quantities of the unstable solid phase in the solution saturated with regard to the stable phase. The tensile strength of set cement depends to a large extent on a film of solution adsorbed upon the precipitated particles of the stable solid phase.

R. TRUSZKOWSKI.

Gypsum. P. P. BUDNIKOV (Pit and Quarry, 1928, 16, 72–76).—Sulphuric, nitric, hydrochloric, and hydrofluoric acids are accelerators, whilst phosphoric, lactic, boric, formic, acetic, and citric acids are retarders of the setting of plaster of Paris. Salts of the univalent metals are positive catalysts; bivalent metal sulphates differ but slightly in catalytic activity. The mechanism of the effects is discussed.

CHEMICAL ABSTRACTS.

Methods for obtaining high tensile strength of gypsum cement. T. MAEDA (Sci. Papers Inst. Phys. Chem. Res. Tokyo, 1928, 8, Suppl., 8–9).—The tensile strength of gypsum cements is doubled by the addition of ethyl or methyl alcohol to the mixture of gypsum and water. Gypsum warmed to 120° in an oil-bath so that the resulting product has the composition $\text{CaSO}_4 \cdot 0.24\text{H}_2\text{O}$, yields a cement of greater tensile strength than without preheating, whilst a product of about the same water content obtained by heating to a higher temperature yields a product of inferior strength.

R. TRUSZKOWSKI.

PATENTS.

Manufacture of cement. AMME-LUTHER WERKE BRAUNSCHWEIG DER “MIAG” MÜHLENBAU U. IND. A.-G. (B.P. 284,294, 27.1.28. Ger., 27.1.27).—Coloured (e.g., ferruginous) raw ground materials are mixed with suitable amounts of decolorising substances, such as borax, together with a flux (but excluding alkali or alkaline-earth chlorides), and then sintered, or melted and granulated, and further treated to form cement. By the further addition of a reducing agent, such as coke, to the raw materials, or by calcining in a reducing atmosphere, iron compounds are reduced and the mass is magnetically treated, after coarse and fine grinding, to remove the metal. The raw materials may be treated individually by the process before mixing.

A. COUSEN.

Manufacture of cements rich in alumina. A. HASSELBACH, Assr. to G. POLYSIUS (U.S.P. 1,677,842, 17.7.28. Appl., 27.9.24. Ger., 19.2.24).—The raw material, together with a small quantity of fluorspar, is burned in a rotary kiln at a temperature below the m.p. now used without fluorspar.

H. ROYAL-DAWSON.

Rotary [cement etc.] kiln. F. HURLBUTT (B.P. 293,331, 24.3.27).—The tapered blocks or bricks forming the lining of a rotary kiln for burning Portland cement, lime, soda, etc. are provided with a rebate along one of the edges of the wider end, and a corresponding projection at the opposite edge of the same end. The bricks thus lock together at the outside, and are prevented from falling into the kiln even when badly eaten away.

A. COUSEN.

Cold bituminous paving composition. O. H. BERGER, Assr. to W. P. McDONALD CONSTRUCTION CO. (U.S.P. 1,672,361, 5.6.28. Appl., 21.8.25).—A hard asphalt having a penetration not greater than 4 (46.1°, 100 g., 5 sec.) is heated with an equal quantity of a petroleum oil (e.g., topped crude oil) and mixed with the usual aggregates.

F. G. CLARKE.

Mixing metallic salt [copper sulphate] with asphaltic compounds. F. L. CARSON (U.S.P. 1,677,272, 17.7.28. Appl., 13.5.26).—Copper sulphate solution in excess is mixed with a small quantity of bitumen, and after evaporating off the water the mixture is incorporated with excess of hot bitumen.

H. ROYAL-DAWSON.

Cementitious material of cellular texture. G. B. HINTON (B.P. 294,041, 24.11.27).—See U.S.P. 1,657,716; B., 1928, 266.

Preserving and protecting vegetable substances [wood]. L. P. CURTIN (B.P. 293,908, 19.4.27).—See U.S.P. 1,659,135; B., 1928, 299.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Electric furnace cast iron. C. E. WILLIAMS and C. E. SIMS (U.S. Bur. Mines Tech. Paper 418, 1928, 48 pp.).—Iron having about twice the strength of cupola iron can be made in the electric furnace. A tensile strength of more than 40,000 lb./sq. in. is common together with transverse strengths of 5000–6000 lb. on standard bars of grey iron. The advantages in

strength and toughness are due largely to the density and fine-grained structure of the metal, which, although whiter than cupola iron of equal carbon content, is soft and is easily machined. Virtually any kind of ferrous scrap may be used in the electric furnace, and when producing synthetic cast iron from steel scrap the power consumed ranges from 500 to 700 kw.-hours per ton, little variation occurring between the basic and acid processes. Considerable value attaches to being able to maintain the molten metal in the furnace until proper adjustments in composition are made and the metal is cast accurately as desired.

C. A. KING.

Cementation of steels by special manganese alloys. J. CURNOT (Compt. rend., 1928, 187, 298—300).—The characteristics of the cementation obtained from hard, semi-hard, and soft steels with ordinary spiegel, ferromanganese (76.19% Mn, 6.52% C), and ferromangano-silico-aluminium (19.25% Mn, 20.64% Si, 10.34% Al) are described, and hardness values are given. The best cementations were obtained when about 2.5% each of ammonium chloride and of alumina were mixed with the cement, and the heating carried out for 4 hrs. at 900—1050°, but the penetration was progressively less marked for successive cementations. Soft steel cemented with ferromangano-silico-aluminium is readily attacked by concentrated hydrochloric acid and dilute (10%) mineral acids, but only slightly by concentrated sulphuric and nitric acids.

J. GRANT.

Creep in five steels at different temperatures. H. J. FRENCH, H. C. CROSS, and A. A. PETERSON (U.S. Bur. Mines, Tech. Papers No. 362, 1928, 22, 235—267; cf. B., 1926, 57).—Creep tests were made on a low-carbon structural steel, a high-chromium steel, a chromium-molybdenum structural steel, high-speed steel, and a high-chromium, high-nickel austenitic steel, and the results are summarised in creep charts in which are shown the relations between stress, temperature, elongation, and time for each steel at temperatures ranging from 20° to 730°. By means of these charts the approximate determination of the stresses which permit life of different durations with different total elongations may be determined. In the temperature range within which strain-hardening takes place, the relation is shown between temperature and the stresses producing different initial elongations which are followed by a practical cessation of creep. At temperatures above those at which strain-hardening occurs the relation is shown between temperature and the per cent. elongation in 1000 hrs. Correlation of creep tests with short-time tension tests showed that, when using accurate equipment, the proportional limit was in the range of stresses which could be sustained for long periods with small amounts of deformation; as the temperature was raised, however, the proportional limit showed a tendency to become higher than the allowable creep. Steels which withstand the highest stresses at atmospheric or slightly elevated temperatures do not necessarily show superiority at higher temperatures. The load-carrying ability begins to drop rapidly at 315—425°. Between 595° and 730° the high-speed steel and the high-chromium, high-nickel steel gave the best results, whilst the chromium-molybdenum steel gave the

poorest; this is probably due to the fact that it was not structurally stable at 650°, oxidation being accompanied by decarburisation and grain growth. The best resistance to oxidation in air was shown by the high-chromium, high-nickel steel and the high-chromium steel.

M. E. NOTTAGE.

Internal structure of chromium steels. E. MAURER and H. NIENHAUS (Stahl u. Eisen, 1928, 48, 996—1005).—The work of earlier investigators on the composition of residues obtained by dissolving chromium steels in acids or by electrolysis is summarised and critically reviewed; it is concluded that the existence of the carbides Cr_3C_2 , Cr_4C_2 , and Cr_5C_2 is definitely established, but that Cr_4C is doubtful. Measurements of the electrical resistance of two series of chromium steels provided evidence of the presence of chromium carbides but gave no indication of their composition. The transformation points in a series of 22 chromium steels containing up to 35% Cr have been determined by thermal and dilatometric analysis and by magnetic measurements. The limits of the γ field at high temperatures are 17% Cr with 0% C, 25% Cr with 0.55% C, and 28% Cr with 1% C. In alloys with 0.55% C the Acl and Ac2 lines intersect at 2% Cr and in alloys with 1% C at 2.5% Cr. The slope of the Acl line is steeper the higher the carbon content for alloys containing up to 10% Cr. The Ac2 line falls in a smooth curve from 770° with 10% Cr to 600° with 32% Cr in alloys with 0.55% C, and to 600° with 29% Cr in alloys with 1% C. No indications of transformations due to chromium carbides were found.

A. R. POWELL.

Self-hardening property of chromium steel. I. OBINATA (Ryojun Coll. of Eng., 1928, 1, 145—166).—This investigation deals with the thermal data and microstructure of a series of chromium steels (Cr 7—18%) in which the ratio Cr/C varied from 50 to 75. In the annealed condition, the Acl carbide change point was raised and the Ar1 point was lowered by the addition of chromium. The temperature of the A2 magnetic change point was lowered by about 7° for each 1% Cr added, and was not affected by annealing or quenching. In the annealed specimens the stable structure, which was only attainable with a very slow rate of cooling, was pearlitic, the two phases probably consisting of a solid solution of iron and chromium and a carbide of chromium containing a little chromium in solid solution, the temperature of the A1 point varying with the composition of the solid solution. Within certain ranges of temperature, the γ -phase exists throughout the whole series of quenched specimens. The Acl point was lower and the quantity of heat absorbed was less in the quenched than in the annealed specimens, the difference increasing with the quantity of chromium, and both being unaffected by the time of soaking. A theory of the self-hardening property based on the experimental results is discussed in detail. It is suggested that these steels contain a carbide of chromium of unknown formula which dissociates above the Acl point. The dissociation and re-formation of this carbide is comparatively rapid at high temperatures, but the temperature coefficient of the reaction velocity is so great that the carbide cannot re-form below the normal A1 point; hence it may be

suppressed by quenching. The depression of the carbide change point on cooling which imparts the self-hardening property is due to the increase in viscosity of the steel produced by the addition of chromium which retards the deposition of the re-formed carbide from the super-saturated solid solution. M. E. NOTTAGE.

Separation of molybdenum from vanadium in steel. J. KASSLER (Z. anal. Chem., 1928, 74, 276—289).—The method depends on the fact that in the presence of ferrous chloride vanadium is precipitated completely with the iron by the addition of an excess of sodium hydroxide, whereas the molybdenum dissolves as sodium molybdate. For steels with less than 3% Mo 4 g. are dissolved in hydrochloric acid and potassium chlorate; the solution is boiled to expel chlorine, diluted, filtered, treated with 25 c.c. of 20% sodium sulphite solution to reduce part of the iron to ferrous chloride, and poured in a thin stream into 120 c.c. of hot 24% sodium hydroxide solution. An aliquot part is filtered and the molybdenum determined as lead molybdate in the usual way. For steels with more than 3% Mo 1 g. is dissolved in 50 c.c. of hydrochloric acid (*d* 1.12) and 25 c.c. of 5% potassium chlorate solution and the solution is evaporated to a syrup. After dilution and filtration, 3 g. of ferrous chloride are added and precipitation of the iron is effected as before. Tungsten interferes in both procedures. A. R. POWELL.

Corrosion by acids and rusting of iron containing copper. S. S. STEINBERG (Ann. Inst. Polyt. Ural, 1927, 6, 73—82).—Tests were made with sheets containing from 0.13 to 1.21% Cu and with 10 and 20% solutions of sulphuric, hydrochloric, and nitric acids. Iron with 0.25—5% Cu dissolves in sulphuric acid solutions 8—10 times as slowly as ordinary iron, and in hydrochloric acid solutions twice as slowly. These effects are regarded as due to the fact that the acid dissolves the iron first, so that the copper becomes concentrated in an atomic-disperse state in the surface layers of the metal. Removal of the surface layer is followed by rapid initial attack of the iron by the acid, the metal gradually increasing in its resistivity. Experiments on rusting in air gave widely varying results, but the mean figures give for the ratio between the resistances of iron containing and free from copper the value 1.5:1. With metal immersed in water or in 1% sodium chloride solution the presence of copper does not increase the resistance. When plates of copper-free and copper-containing iron are immersed in water after removal of the scale by treatment with dilute sulphuric acid, the potential of the former is about 0.020 volt lower than that of the latter. If iron containing copper is heated in oxidising gases, copper is observed beneath the surface scale, the iron being oxidised in preference to the copper.

T. H. POPE.

Influence of the mould and the degree of deoxidation on the crystallisation of [steel] ingots cooled without disturbance. F. BADENHEUER (Stahl u. Eisen, 1928, 48, 713—718, 762—766).—The shape of the mould used in making chill castings of steel has much more effect on the soundness of the ingot than has the thickness of the walls. The tendency to the formation of secondary pipes is reduced by using a mould tapering

towards the top end or by casting with a large waste-head. In the centre of all cylindrical castings there is always a more or less extensive zone with a characteristic globular structure. This zone extends practically throughout the ingot when a small quantity of deoxidiser is added to the steel just prior to casting, but with larger amounts of deoxidiser, especially with aluminium, the zone becomes more confined. Just below its apex V-shaped segregations of phosphides, sulphides, and non-metallic impurities are generally visible. The remainder of the ingot has a dendritic structure with a definite crystallite orientation; in this zone normal segregation of impurities takes place, whereas in the globular zone, inverse segregation is usual. These phenomena are attributed to variations in the gas content of the steel due to the use of deoxidisers. A. R. POWELL.

Influence of phosphorus on strength properties of cast iron pipe. J. T. MACKENZIE (J. Amer. Water Works' Assoc., 1928, 19, 747—759).—Up to 0.8% P improves the quality of cast iron pipe by increasing the fluidity of the molten metal, but beyond 1% its stiffening and embrittling effects outweigh this advantage, and strength and resilience decrease rapidly. C. JEPSON.

Effect of temperature on the solubility of iron oxide in iron. C. H. HERTY, JUN., and J. M. GAINES, JUN. (Amer. Inst. Min. Met. Eng. Tech. Bull., 1928, No. 88, 13 pp.).—At 1600°, the normal open-hearth tapping temperature, the solubility of ferrous oxide in iron is 1.36%, at 1900° 2.02%. Thus the oxygen is present in molten iron as a compound, and not as a gas; the constancy of the distribution coefficient between the metal and slag (ferrous oxide, with or without lime) indicates that this compound is ferrous oxide. The rate at which carbon disappears in open-hearth elimination depends on the rate of diffusion of ferrous oxide from the slag into the metal, and this depends on the saturation value of ferrous oxide under the slag in question.

CHEMICAL ABSTRACTS.

Behaviour and necessary properties of dolomite for steel works. O. JACOBS (Stahl u. Eisen, 1928, 48, 993—995).—For the lining of basic converters for the Thomas process the dolomite used should be free from carbon dioxide, and should be so burnt that the particles are sufficiently porous to absorb completely the tar oils from the binder during the operation of lining the furnace. A dolomite with 5—8% SiO₂ + Fe₂O₃ + Al₂O₃ after burning is more suitable than a purer product as its sintering temperature is lower, and therefore an efficient agglomeration of the grains takes place during the heating of the first charge. Other things being equal, an amorphous dolomite gives better results than a crystalline variety. The lime used for slagging the phosphorus in the iron should be as free as possible from magnesia, which produces a viscous slag and causes a more rapid wear of the lining. For the basic open-hearth furnace a dolomite low in silica and high in magnesia gives the best results in the fire-bridge, and for the hearth better results are obtained with burnt dolomite containing 28—40% MgO, 3—7% SiO₂, and 4—8% Fe₂O₃ + Al₂O₃. The hearth should be tempered with a slag rich in fluxes previous to charging in the iron. Alternatively,

the top layer of the hearth may be made of a sintered mixture of dolomite and old slag in the ratio 4 : 1.

A. R. POWELL.

Iron carbonyl and carbonyl iron. A. MITTASCH (Z. angew. Chem., 1928, 41, 827—833).—Iron pentacarbonyl is produced on the large scale by circulating carbon monoxide under pressure over sponge iron at 150—200° and cooling the gases evolved. It has d_4^{20} 1.453, a surface tension of 22 dynes/cm. and a viscosity of 0.0075 c.g.s. unit at 20°. Its heat of formation (liquid) is 54.2 kg.-cal., latent heat of fusion 3.25 kg.-cal./mol., and heat of combustion 384.5 kg.-cal./mol. In alkaline solution it behaves as a powerful reducing and dechlorinating agent for organic compounds. In pentane solution it combines with bromine forming a yellow unstable compound which rapidly decomposes with the evolution of carbon monoxide and the formation of the reddish-brown compound, $\text{Fe}(\text{CO})_4\text{Br}_2$. Iron pentacarbonyl decomposes at 250° in a hollow vessel heated by radiation yielding a finely-divided iron containing about 1% C. If this is melted in a vacuum high-frequency furnace together with the requisite quantity of pure iron oxide, obtained by combustion of the carbonyl, an exceedingly pure iron of high permeability, low hysteresis, and small wattage loss is obtained.

A. R. POWELL.

Determination of zinc in presence of large percentages of arsenic. L. R. RAYMOND (Chemist-Analyst, 1928, 17, 6).—The ore (0.5 g.) is heated with sodium sulphate (2 g.), concentrated sulphuric acid (7—10 c.c.), and a small piece of filter paper until all the acid has evaporated and the paper is completely oxidised. The cool mass is disintegrated with warm water (10 c.c.) and boiled to a paste with concentrated hydrochloric acid (20 c.c.); a second evaporation with hydrochloric acid may be necessary to volatilise all the arsenic. Finally concentrated hydrochloric acid (10 c.c.) and ammonium chloride (10 g.) are added, the mixture is heated and diluted to 150 c.c., excess of ammonia and 10 c.c. of hydrogen peroxide are added, the liquid is filtered, and the ferric hydroxide washed with hot ammonium chloride solution; if much iron is present the hydroxide is redissolved and reprecipitated. Hydrochloric acid (7—8 c.c. excess) is added to the filtrate, which is diluted to 400 c.c., copper is precipitated with lead, and the zinc titrated with ferrocyanide.

CHEMICAL ABSTRACTS.

Determination of nickel with α -benzildioxime in presence of chromium compounds. F. G. GERMUTH (Chemist-Analyst, 1928, 17, 3, 7).—Contamination of the nickel precipitate by chromium is avoided by the addition of 0.25 g. of potassium hydrogen tartrate and 0.2 c.c. of 10% cupric ammonium chloride solution.

CHEMICAL ABSTRACTS.

[Mechanical properties and crystal orientation of] copper and brass. O. BAUER, VON GÖLER, and G. SACHS (Z. Metallk., 1928, 20, 202—208).—The tensile strength of hard-rolled α - and β -brass increases almost linearly with the angle of the test-piece to the direction of rolling; with pure copper and a zinc-copper alloy (2% Zn) the tensile strength falls to a slight minimum when the test piece is taken at an angle of 45° to the direction of rolling. After annealing at 500° the con-

verse holds true; copper has maximum strength at an angle of 45° to the direction of rolling, and the strength of brass falls slightly as this angle increases from 0° to 90°. The elongation of rolled copper annealed for 30 min. at 500° is about 20% in directions parallel to and at right angles to the direction of rolling, but rises sharply to 75% in a direction midway between these. The impact strength (notched-bar test) of cold-worked copper and brass rises sharply with the temperature of annealing above 200° to a maximum at 450—600°, and then falls almost as abruptly in the case of α -brasses, but rises slightly with ($\alpha + \beta$)-brasses. The impact strength of hard-worked copper-zinc alloys rises to a maximum with 85% Cu; after annealing at 600° for 2 hrs. the maximum impact strength is obtained with 85—70% Cu. Characteristic röntgenograms and photographs of fractured test-pieces of hard-worked and annealed copper and brass sheets are reproduced, together with photomicrographs of heat-treated brass.

A. R. POWELL.

Gold-silver-copper alloys. F. E. CARTER (Amer. Inst. Min. Met. Eng. Tech. Pub., 1928, No. 86, 17 pp.).—Physical properties are reported for alloys of gold with silver or copper; zinc is a useful deoxidiser for the 14-carat alloys. Increase of density on rolling indicates gas inclusions.

CHEMICAL ABSTRACTS.

Platinum metals and their alloys. F. E. CARTER (Amer. Inst. Min. Met. Eng. Tech. Pub., 1928, No. 70, 24 pp.).—The treatment of platinum ores is described and physical properties of platinum metals are tabulated. Applications of the metals are discussed.

CHEMICAL ABSTRACTS.

Dissolution of aluminium and its alloys in acids. X. WACHÉ (Rev. Mét., 1928, 25, 331—346).—The dissolution of commercial aluminium in hydrochloric acid occurs in two stages, a rapidly increasing rate of dissolution in the first stage being followed by a more or less steady rate after the "period of induction." Rise of temperature or increase of concentration of acid increases the rate of attack, though the more important factor is the quantity and distribution of iron and silicon in the metal. The more highly disseminated impurity causes more rapid dissolution of the alloy, and the effect of heat-treatment is reflected entirely by the greater or lesser homogeneity of the resultant alloy.

C. A. KING.

Mechanical properties of aluminium alloys. R. KARNOP and G. SACHS (Z. Physik, 1928, 49, 480—497).—Data are given for the tensile strength and hardness of single crystals of the following aluminium alloys under various conditions of mechanical deformation: Al 99%, Mg 0.5%; Al 95%, Cu 5%; Al 94%, Mg 0.5%, Cu 5%. The results are discussed with reference to the crystal structure of the specimens before and after deformation.

R. W. LUNT.

Determination of lead as chromate in Babbitt metal, and determination of copper by Walker and Whitman's method. G. SCHESTAKOV (J. Chem. Ind. [Russia], 1927, 4, 907—908; Chem. Zentr., 1928, i, 1307—1308).—The alloy containing lead, copper, tin, and antimony is dissolved in aqua regia, citric acid is added, and the liquid is neutralised with ammonia; it is then acidified with acetic acid, boiled, and the lead is precipitated with potassium dichromate, the copper in

the filtrate being precipitated with hydrogen sulphide or dextrose.

A. A. ELDRIDGE.

Resistivity of chromium-plated metals to the action of chemical reagents. T. MURAKAMI (J. Soc. Chem. Ind. Japan, 1928, 31, 132—136).—Commercial chromium is ordinarily in the passive state, and is highly resistant to acids and alkalis, except hydrochloric acid. The new surface obtained by breaking the commercial metal is no longer resistant to acids. Moreover, passive chromium in contact with zinc, iron, or some other metals in acids becomes active and is not resistant to acids. The chromium film of chromium-plated metals is generally pin-holed, and the plated metals are easily dissolved by acids. The fresh chromium film is active and not resistant to acids, but after some time it becomes passive. The chromium film prepared by the method of Kato and the author (B., 1928, 576) is resistant to many reagents and gases. The length of time after plating and pin-holes in the chromium film are the two important factors for testing the resistivity of chromium-plated metals.

K. KASHIMA.

Autogenous welding with coal gas. A. MÜLLER and B. BIBUS (Gas- u. Wasserfach, 1928, 71, 566—569).—Coal gas with a calorific value of 4230 kg.-cal./m.³ gives when burnt with oxygen a flame temperature of about 1980° compared with 3200° for the oxy-acetylene flame. The useful heat generated is about 3850 and 2600 kg.-cal./m.³, respectively, for acetylene and coal gas, and the oxygen-consumption 1 and 0.45 m.³/m.³, but the cost of operation of the oxy-coal gas burner is only about $\frac{1}{3}$ — $\frac{1}{2}$ that of the oxy-acetylene burner. On the other hand the oxy-acetylene flame with a standard burner will weld a sheet of metal 4—5 times as thick as that which can be welded by an oxy-coal gas flame from the same burner, so that for heavy work the oxy-acetylene flame is not only much more efficient but also more economical. For light work with aluminium or brass the cost of the oxy-coal gas burner is 5—15% less than that of the oxy-acetylene burner.

A. R. POWELL.

Trend of flotation. A. J. WEINIG and I. A. PALMER (Quart. Colorado School Mines, 1928, 23, [2], 1—90).—A second, revised edition giving a complete review of modern American practice in flotation with a bibliography up to January, 1928 (cf. B., 1927, 192).

A. R. POWELL.

Danger of mercury and amalgam dental fillings. A. STOCK (Z. angew. Chem., 1928, 41, 663—672).—The use of mercury in dental fillings (copper or silver amalgam) is especially deprecated as dangerous both to dentists and patients, as air containing 0.001 mg. of mercury per m.³ is injurious to health.

C. IRWIN.

Absorption of atmospheric moisture by molten metals. P. KOSOVSKI (Messager russe ind. mét., 1927, 50—51).—With metals of low m.p. (e.g., tin), relatively large amounts of water may sometimes be found in the cavities formed by shrinking on cooling. With metals of high m.p. (e.g., iron or copper) hydrogen is formed and cracks may develop on rolling. The casting of iron or copper should be carried out in as dry an atmosphere as possible.

CHEMICAL ABSTRACTS.

Dilatometry of light metals. M. HAAS (Z. Metallk., 1928, 20, 283—285).—A description of technique.

Tungstic oxide. GERO and IREDELL.—See VII.

PATENTS.

Manufacture of iron [castings]. M. G. WOODMAN, Assr. to D. G. FISHER and V. F. SHANTZ (U.S.P. 1,677,420, 17.7.28. Appl., 16.5.27).—Between 1 and 3 lb. of salicylic acid are added per ton of molten iron before casting.

F. G. CLARKE.

Multiple-hearth calcining furnace. R. O. PIKE (U.S.P. 1,674,919, 26.6.28. Appl., 19.11.23).—The combustion chamber is situated between two ore hearths, and is surrounded by an annular air chamber from which air is supplied to the combustion chamber through radial passages in the hearth above and through fuel burners.

B. M. VENABLES.

Operation of open-hearth furnaces. A. L. STEVENS, Assr. to A. L. STEVENS CORP. (U.S.P. 1,677,664, 17.7.28. Appl., 16.5.27).—An open-hearth furnace is operated with a neutral atmosphere in the furnace chamber at atmospheric pressure.

C. A. KING.

Manufacture of steel in the Siemens-Martin furnace. F. SIEMENS A.-G., R. DURRER, F. C. SIEMENS, and A. SPRENGER (B.P. 293,326, 24.12.26).—In order to produce a protective layer of gas free from hydrogen or hydrocarbons over the metal in the furnace after melting down, gas produced in a "tapping" producer from coke without the use of steam is introduced through ports under the gas mixing chamber, and is directed on to the surface of the metal. Various modifications in the design of the furnace chamber are indicated.

C. A. KING.

Increasing the permeability of silicon steel. J. C. KARCHER, Assr. to WESTERN ELECTRIC CO. INC. (U.S.P. 1,677,139, 17.7.28. Appl., 19.9.24).—The steel is heated to 1000—1250° and cooled at not more than 10° per min., reheated to 550—800°, and cooled at a rate slower than quenching in water but faster than quenching in air.

F. G. CLARKE.

Producing permanent magnets. L. E. HOWARD and A. D. POTTS, Assrs. to SIMONDS SAW & STEEL CO. (U.S.P. 1,677,674, 17.7.28. Appl., 9.10.26).—The magnet consists of tungsten steel containing 0.05 to 1% V.

H. ROYAL-DAWSON.

Bonding metal. F. C. MATHISON, Assr. to C. L. REDFIELD (U.S.P. 1,677,354, 17.7.28. Appl., 2.9.24).—An iron or steel body is coated with a deposit of copper, then with a thin film of tin, and finally with the lead-base bearing metal by the babbiting process.

H. ROYAL-DAWSON.

Extraction of metals from ores. P. R. BLAWIE (B.P. 293,340, 2.4.27).—The ore, particularly cassiterite or other tin ore, after concentration is calcined in compressed heated air and, while still hot, is reduced wholly or partially to the metallic state by means of a reducing gas heated under pressure. The reduced ore is concentrated by flotation and then washed in molten metal, the operation being conducted in several stages at different temperatures to remove the bulk of the metal from the gangue. The remainder of the metal may be recovered from the residual material by causing the particles to slide down a channel-shaped electrode in an

electrolytic cell and then treating them with an acid or other solvent or a gas, such as chlorine.

M. E. NOTTAGE.

Ore concentration. B. R. TUNISON (U.S.P. 1,677,416, 17.7.28. Appl., 9.2.24).—An oily, sulphur-containing constituent extracted from crude petroleum by means of caustic alkali is used as froth-flotation agent.

F. G. CLARKE.

Manufacture of highly porous, coherent lead aggregates. I. G. FARRENIND. A.-G. (B.P. 270,763, 9.5.27. Ger., 7.5.26).—Porous aggregates of lead suitable for accumulator plates, or for filtering or adsorption purposes, are formed from a homogeneous alloy of lead and an alkali or alkaline-earth metal, or a mixture of lead and an organic compound, from which the added material is extracted by suitable methods. C. A. KING.

Metal powder and method of producing it. E. KRAMER, and HARTSTOFF-METALL A.-G. (HAMETAG) (B.P. 293,609, 11.10.27).—Metal powders or granules having a grain dimension of 0.5–0.01 mm. consist of granules of hollow or bowl shape. The powder as produced in this manner is particularly adapted for making small pressings, e.g., dynamo brushes, current collectors, etc.

C. A. KING.

Removing gases from and cleaning the surfaces of metals in vacuums by electrical means. J. VON BOSSE (B.P. 286,632, 12.4.27. Ger., 7.3.27).—The metals are used in the form of electrodes in an evacuated space and a current of a few hundred to a few thousand volts is applied.

M. E. NOTTAGE.

Mercury condenser. E. K. PETERS (U.S.P. 1,672,499, 5.6.28. Appl., 10.1.27).—The condenser comprises a tower packed with sponges, which rest upon perforated partitions held by a vertical, central support. The latter enables the entire packing to be withdrawn as a unit, after removal of the top of the tower. The mercury vapour is sucked downwards through the tower, and the sponges are sprayed with water.

F. G. CLARKE.

Alloy. A. C. G. GWYER and H. W. L. PHILLIPS, Assrs. to BRIT. ALUMINIUM CO., LTD. (U.S.P. 1,676,856, 18.7.28. Appl., 26.8.26. U.K., 19.7.24).—See B.P. 243,405; B., 1926, 132.

Determining composition of copper and zinc alloys. M. POLANYI and S. VON BOGDANDY (U.S.P. 1,676,709, 10.7.28. Appl., 25.2.27. Ger., 24.3.26).—See B.P. 268,306; B., 1927, 784.

Mixing apparatus particularly for amalgamating or alloying metals. B. D. WARNE and S. CALVER (B.P. 294,276, 21.4.27).

Lacquering metals (B.P. 283,162).—See XIII.

XI.—ELECTROTECHNICS.

Pressure electrolysis of water. J. E. NOEGGERATH (Chem. Met. Eng., 1928, 35, 421–423).—The principal difficulty in pressure electrolysis, whereby compressors can be eliminated, is that of economically maintaining the gases in a sufficiently pure state. The author electrolyses potassium hydroxide solution in tubular cells with axial nickel cathodes working at 180 atm. A compensating line of large capacity is connected to both compartments, so that differences in pressure

between the two will not alter the electrolyte levels. Such cells may find employment for storage of energy, the hydrogen to be used in motors, or in locomotives on branch lines of electric systems. Dissociation requires lower voltage, and hence less power consumption with increasing pressure, and, in view of the simple construction of the cells, it is claimed that they can replace ordinary cells for the production of electrolytic hydrogen for chemical purposes.

C. IRWIN.

Ageing phenomena in quartz mercury-vapour lamps. W. MEYN (Z. wiss. Phot., 1927, 25, 345–353).—Devitrification of the quartz plays only a secondary part in the ageing effect, for ageing takes place in lamps even where devitrification is absent. In studying the ageing effect the band spectrum of Stark and the continuous spectrum of Küch and Retschinsky are of minor importance, hence the line spectrum only was investigated, at wave-lengths less than 4000 Å. A Hilger quartz spectrograph was used, fitted with an arrangement which brought the whole spectrum sharply into focus on the same plate. The decrease of intensity with age was measured by adjusting the time of exposure till a given line showed a standard intensity. The fall of intensity is due not to the presence of foreign gases (e.g., the inert gases) which some have supposed can diffuse through the quartz at high temperature, but to a black deposit which appears on the inner surface of the quartz and consists of two parts, one of which can be removed by rubbing or by the action of acids, and the other penetrates to a depth of 0.1 mm. into the quartz itself. This latter layer may be due to the presence of very finely-divided mercury, or, more probably, to the separation of small particles of elementary silicon formed under the influence of the ultra-violet radiation and the high temperature.

S. J. GREGG.

PATENTS.

Magnetic cores. STANDARD TELEPHONES AND CABLES, LTD., Asses. of G. W. ELMEN (B.P. 277,289, 12.4.27. U.S., 11.9.26).—Finely-divided particles of an alloy capable of having imparted to it an initial permeability greater than that of iron, and very sensitive to mechanical strains, are mixed, under pressure, with an insulating material capable of retaining its insulating and binding properties at annealing temperatures of at least 800°; the mixture is heat-treated at 450–800° to develop magnetic permeability. The heat treatment may precede the compression. The magnetic element may consist of an alloy of nickel and iron, and the insulating particles may be intermixed with a lubricant.

M. E. NOTTAGE.

Preparing electrical resistances from ceramic material. M. HAUSER (B.P. 293,652, 26.4.27).—To the ceramic material containing silicon or ferrosilicon as conductor with or without other metallic admixtures, substances containing boric acid in the free or combined state are added whereby the temperature coefficient of the resistance may be varied from weakly negative to strongly positive. The resistances are fired in a reducing or a non-oxidising atmosphere.

M. E. NOTTAGE.

Lead aggregates (B.P. 270,763). **Metal powder** (B.P. 293,609). **Cleaning metals** (B.P. 286,632).—See X.

XII.—FATS; OILS; WAXES.

Classification of fats. I. W. HALDEN (Chem. Umschau, 1928, 35, 55—56).—The following classification of fats and oils is suggested: (i) From marine and cold-blooded ("poikilothermal") animals; iodine value above 100. (ii) From warm-blooded ("homoiothermal") animals (excluding marine animals); iodine value below 100. E. LEWKOWITSCH.

Behaviour of fats and oils in ultra-violet light. M. HAITINGER, H. JÖRG, and V. REICH (Z. angew. Chem., 1928, 41, 815—819).—The fluorescence colours under ultra-violet light of several oils and fats, pure and in solution, are recorded, with reference to the detection of adulterants in butter, lard, cacao butter, olive oil, etc. A characteristic zoned fluorescence is noted when filter paper partially immersed in oil or oil solution is saturated by capillary rise of the liquid. E. LEWKOWITSCH.

Hexabromide of elæostearic acid. K. H. BAUER and E. ROHRBACH (Chem. Umschau, 1928, 35, 53—54).—By bromination of α -elæostearic acid in light petroleum solution in the presence of ultra-violet light, a solid hexabromide, m.p. 139—141°, was obtained and a dark-red oily mixture of substituted bromo-acids. E. LEWKOWITSCH.

Oxidation processes of drying oils. S. MERZBACHER (Chem. Umschau, 1928, 35, 173—176).—Among the products of lixiviation and saponification of linoxyn formic, propionic, and azelaic acids were identified, and a mixture of crude acids was obtained containing an acid (caprylic or pelargonic) of m.p. 10°. E. LEWKOWITSCH.

Detergent experiments on cotton. R. M. CHAPIN (Oil and Fat Ind., 1928, 5, 208—212).—The detergent power of soap solutions was tested on cotton soiled with ointments of lamp-black and lard, vaseline, and mineral oils (cf. B., 1928, 417 for cotton soiled with non-oily carbon black). Of pure sodium laurate, myristate, palmitate, stearate, and oleate, the last-named was most powerful up to 40°, being surpassed by palmitate at 60° and by stearate at 80°. Addition of sufficient fat solvent (e.g., carbon tetrachloride) enhanced the detergent power. In parallel with the author's results on emulsification, excess of fatty acids increased and excess of alkali diminished the detergent power. E. LEWKOWITSCH.

Avocado [pericarp] oil. G. S. JAMIESON, W. F. BAUGHMAN, and R. M. HANN (Oil and Fat Ind., 1928, 5, 202—207).—Oil obtained from the dehydrated pulp of the avocado fruit by expression or extraction is dark green by transmitted, and red by reflected light; it has d_{25}^{25} 0.9132, n_D^{25} 1.47, acid value 2.8, saponif. value 192.6, unsaponif. matter 1.6%, iodine value (Hanus) 94.4, acetyl value 9.2, Reichert-Meissl value 1.7, Polenske value 0.2, iodine value of unsaturated acids 101.2. Separation of the acids by the lead salt-ether method, and fractionation of the methyl esters, gave the composition of the oil as glycerides of: oleic 77.3, linoleic 10.8, myristic trace, palmitic 6.9, stearic 0.6%, and arachidic acids trace. The pulp (68—85% of the fruit) contains at least 70% of the oil, which can be partially bleached by fuller's earth, shows good keeping properties, and is suitable for the manufacture of hard soap. E. LEWKOWITSCH.

Malvaceæ. III. Hibiscus manihot, L. J. PIERAERTS (Mat. grasses, 1928, 20, 8138—8139).—The seed contains water 10.80, total ash 4.56, water-soluble ash 2.68, total nitrogen 3.51, crude protein 23.34, ether extract 13.42, crude fibre 24.95, pentosans 11.44, phosphorus pentoxide 1.07%; the alkalinity of the total and soluble ash is 24.88 and 60.64% (as potassium carbonate), respectively. The oil, extracted with light petroleum of b.p. <60°, had d_{20}^{20} 0.9194, acid value 6, saponif. value 197, iodine value 103, Crismer value (99.6% alcohol) 62°, n_D^{23} 1.4695, unsaponifiable residue 0.92%, Hehner value 95.80%; Halphen reaction positive. The Millian-Becchi reaction gives strong reduction with formation of a silver mirror.

CHEMICAL ABSTRACTS.

Isomerism among unsaturated fatty acids. EIBNER and others.—See XIII. Maize oil. KRIZKOVSKY.—See XVII. Acids in butter fat. VIRTANEN. Starch in oily seed-kernels. GRIEBEL.—See XIX.

PATENTS.

Treatment of oils or fats or mixtures of the same or fatty acids for the production of sulphuric acid compounds. E. C. R. MARKS. From CHEM. FABR. STOCKHAUSEN & Co. (B.P. 293,480, 6.4.27).—After sulphonation of the oils the valuable fatty acid sulphates are separated from the unattacked oil by treatment with a neutral inert solvent, e.g., trichloroethylene. Further, the oil etc. dissolved in such a solvent may be subjected to sulphonation.

E. LEWKOWITSCH.

Preservation and transportation of oils and fats. A. NYROP (B.P. 293,342, 4.4.27).—By treating raw oleaginous vegetable material in emulsifiers with water and a film-producing material, e.g., "albuminates" or carbohydrates, and subsequent spray-drying, a paste or powder is produced which can be conveniently transported, will not melt at comparatively high temperatures, and from which the oil can be recovered for use. E. LEWKOWITSCH.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Accelerating the drying of paints and varnishes. H. A. GARDNER (Amer. Paint and Varnish Manufs.' Assoc., Circ. 335, July, 1928, 592—594).—Preliminary details are given of experiments on the rapid drying of oil paints and varnishes when exposed to a carbon arc at 55—60°. The drying times were reduced to one third or less of those required in a dark room at the same temperature. Apparatus employing lamps suitable for drying newly painted or plastered walls is described. S. S. WOOLF.

Inhibition of mildew on paints. H. A. GARDNER (Amer. Paint and Varnish Manufs.' Assoc., Circ. 335, July, 1928, 595—597).—Mildew formation on paints exposed in climates of high humidity and temperature is inhibited by the use of zinc oxide primers and the introduction of tung oil spar varnish or resins into the finishing coat, a firm hard surface being desirable. S. S. WOOLF.

Methods of testing suitability of paints, varnishes, and lacquers for aeronautical purposes. E. W. J. MARDLES (J. Oil and Colour Chem. Assoc., 1928, 11,

230—253).—The use of paints etc. for protection of wood and metal parts of aircraft is discussed generally, and a number of results indicating the influence of weight of film on protection afforded are quoted. It is shown that two coats are considerably better than one, and three coats slightly better than two, whilst further coats do not increase the protective value to any appreciable extent, and might actually be deleterious. Methods are described for determining various film properties, *e.g.*, scratch hardness, adhesion (bending test), resistance to erosion, resistance to water, oil, and petrol, "dope" solvents, etc. (mechanical rubbing test), drying times, and gloss, and typical results are tabulated.

S. S. WOOLF.

Protective coatings for duralumin and other aircraft alloys. H. A. GARDNER (Amer. Paint & Varnish Manufs.' Assoc., June, 1928, Circ. No. 330, 482—508).—Details of the exposure tests of various types of paint on duralumin etc. begun in 1926 (*cf.* B., 1927, 683) are recapitulated and the results of a recent and final inspection are tabulated. Panels giving superior service were those coated with a zinc chromate paint, an iron oxide-zinc chromate paint, a zinc chromate primer followed by high-grade aluminium paint, and a lampblack paint. Various protective treatments of these light-weight alloys (other than painting) are described.

S. S. WOOLF.

Paints for metal. H. A. GARDNER (Amer. Paint & Varnish Manufs.' Assoc., June 1928, Circ. No. 331, 510—568).—A dissertation on protective paints for iron, steel, copper, and for galvanised, tinned, and other metal surfaces. The individual characteristics of the pigments employed for this purpose are discussed and the methods of painting and types of paints used on various metals are described. Extended consideration is given to special fields, *e.g.*, marine paints, gas-holder paints, pipe coatings, radiator paints, slushing compounds.

S. S. WOOLF.

Painting tests on panels impregnated with zinc chloride or creosote. H. A. GARDNER (Amer. Paint & Varnish Manufs.' Assoc., June, 1928, Circ. No. 334, 588—598).—The results of exposure tests, begun in Sept., 1926 (*cf.* B., 1927, 585), on various white paints applied to the impregnated panels and examined on May 1, 1928, indicated that freshly creosoted wood may not be satisfactorily painted with white paints made on a linseed oil base, even when various types of primers (red lead, aluminium powder, zinc dust, tin- and aluminium-foil sheets) are applied before the white paint coating. It is, however, considered probable that weathering of the creosoted wood for a year or so will produce a surface that will satisfactorily take paint coatings. Generally satisfactory results were obtained on zinc chloride-impregnated panels, although some darkening is noted.

S. S. WOOLF.

Zinc oxide paints. C. P. VAN HOEK (Farben-Ztg., 1928, 33, 2789—2790).—Straight zinc oxide-linseed oil paints are considered by many users to be deficient in flowing out properties, opacity, adhesion, and elasticity. This is attributable in part to the flocculating tendency of the weakly positively charged pigment, badly flowing masses that retain considerable amounts

of oil being formed. The extra oil necessarily added leads to poor opacity, to poor adhesion on wood (owing to absorption of "free" oil), and to poor weather-resistance of such paints used as primings. Further, the formation of zinc soaps causes undue hardness and lack of elasticity of the film. The addition of 5—10% of stand oil in the grinding process corrects the above faults to some extent.

S. S. WOOLF.

Significance in paint technology of isomerism among higher unsaturated fatty acids and glycerides. A. EIBNER, L. WIDENMAYER, and E. SCHILD (Chem. Umschau, 1927, 34, 312—320).—The different types of isomerism possible in the higher unsaturated fatty acids and glycerides are tabulated, characteristic examples being quoted. The acid isolated from the non-drying parsley seed oil (petroselinic acid) is shown to consist largely of Δ^5 -oleic acid. The shifting of the double linking nearer to the carboxyl end of the chain than in normal Δ^8 -oleic acid manifests itself in the solid condition of the Δ^5 -acid at ordinary temperatures. " γ -Linolenic acid" isolated from primrose oil (*Oenothera biennis*) is shown to be $\Delta^{5:8:11}$ -linolenic acid, the geometrically isomeric α - and β -acids both being $\Delta^{8:11:14}$ -linolenic acids. " δ -Linolenic acid," the presence of which in primrose oil is also indicated, is provisionally accepted as the geometrical isomeride of the " γ "-acid. The " γ "- and " δ "-acids should, therefore, more correctly be denoted as α - and β - $\Delta^{5:8:11}$ -linolenic acids, or, for brevity, symmetrical α - and β -linolenic acids. These should theoretically be slower drying than the $\Delta^{8:11:14}$ -acids, the double linking system being nearer to the carboxyl group, but, since linseed oil exceeds primrose oil in content of the respective linolenic acids, definite conclusions on this point cannot be drawn from the greater drying power of linseed oil. The elæostearic acids, many place-isomerides of which are possible, differ from the linolenic acids by virtue of possessing three conjugated double linkings with consequent increase in drying tendency, but the exact relationship between α - and β -elæostearic acids is not yet established. The presence of dilinolenic-monolinoleic glyceride (previously demonstrated by Schmidinger in a Dutch linseed oil and by Davidson in a linseed oil of unknown origin) is established in samples of Bombay, Riga, and La Plate linseed oils in yields of 36.8, 32.7, and 24.3%, respectively. This glyceride is probably the chief drying principal in linseed oil. A new glyceride, *dilinolenic-mono-oleic glyceride*, m.p. 72—73.5°, was isolated, which may account for all the oleic acid in the oil, and dispose of the fallacious "non-drying of oleic acid in linseed oil" view. The known total content of linoleic acid in linseed oil is not accounted for, and dilinoleic-monolinolenic glyceride is probably present in addition to those mentioned above.

S. S. WOOLF.

Preliminary report of Sub-committee of the Royal Academy on modern pigments and mediums. Feb., 1928. (J. Oil and Colour Chem. Assoc., 1928, 11, 260—269).—A general enquiry into artists' raw materials and the problems met with in painting pictures in oils. The nature of the canvas used and methods of priming are discussed, suggested improvements being the introduction of ramie cloth

in place of linen and cotton canvases, and the substitution of gelatin size by viscose solution. The causes of cracking and lowering of tone of pictures are analysed, factors such as changes in refractive index, changes in colour, and tendency to crack of drying oils, use of unsuitable mediums in the undercoating, transparency and oil absorption of various pigments being considered. In solid under-painting pigments of low oil absorption should be used, since they are least affected by changes in the drying oil and are unlikely to cause cracking. The surface of pictures is best protected by applying a thin coating of a solution of beeswax in turpentine, allowing to dry for some weeks, and polishing. The quality of water-colour paper and the permanency of artists' pigments are briefly mentioned.

S. S. WOOLF.

Use of oil shales in the preparation of ultramarine. B. K. KLIMOV and V. A. LANIN (Trans. State Inst. Appl. Chem., Moscow, 1927, No. 5, 89—105).—The substitution of bituminous shales of the Leningrad area for infusorial earth and tar in the preparation of ultramarine was successful, and even permitted the employment of less sulphur. Experimental yields were normal, but factory results were not good. Suitable shales contained volatile matter 30—46.11, non-volatile organic matter 8—10.65, silica 21.51—26, lime 6—8.36, alumina 5—6.29, moisture 0—4.21%.

CHEMICAL ABSTRACTS.

Large-scale titanium pigment production based on old laboratory process. B. K. BROWN (Chem. Met. Eng., 1928, 35, 427—428).—The plant described has a capacity of 10 tons per day of titanium dioxide working the Blumenfeld process. Ilmenite is treated with sulphuric acid in insufficient quantity to convert all the titanium into sulphate, as the coating of sulphate on the particles in any case makes penetration difficult. The solid reaction mass is agitated with cold water and the unattacked ilmenite returned for further treatment. Metallic iron is added to reduce any ferric salts present, and as much ferrous sulphate as possible crystallised from the clarified solution. Titanium sulphate is hydrolysed from a concentrated solution by a method not indicated, and the titanium dioxide filtered in Sweetland presses. It is washed, dried, and calcined. The finished product contains less than 0.005% of total iron and the pigment made has an oil absorption of about 25. The dioxide can also be prepared as a colloidal dispersion containing 600 g./litre TiO_2 .

C. IRWIN.

Microscopy of body colours. H. WAGNER and J. KESSELRING (Z. angew. Chem., 1928, 41, 833—837).—The use of the microscope in determining the nature of the constituents of natural and artificial pigments is illustrated by several examples. Various colloidal constituents may be identified by their adsorptive power for certain dyes; e.g., kaolin is coloured by basic dyes, colloidal silica by both acid and basic dyes, kieselsol by basic dyes, and hydrated alumina by acid dyes. Suitable dye solutions contain 1% of Brilliant Green, Acid Violet, or Orange II. Crystalline substances, such as fluorspar, calcite, or dolomite, are detected by the polariscopé.

A. R. POWELL.

Lacquer solvents. H. A. GARDNER (Amer. Paint and Varnish Manufs.' Assoc., Circ. 335, July, 1928, 597—606).—Ethyl α -hydroxyisobutyrate and synthetic ethyl lactate (as distinguished from the fermentation product) are recently developed solvents suitable for use in cellulose lacquers—nitrate and acetate. Their chemical and physical properties are detailed.

S. S. WOOLF.

Resin from *Pinus silvestris*. II. Solid constituents. B. ARBUZOV (J. Russ. Phys. Chem. Soc., 1928, 60, 707—719; cf. B., 1927, 971).—The crystalline solid, m.p. 118—130° (33—35% of the total), which separates from the raw resin on keeping has $[\alpha]_D$ —102.23° to —60.47° in alcohol (in benzene about 20° less). The dispersion was almost constant, $[\alpha]_D/[\alpha]_C = 2.37$ —2.39, closely similar to that of the solid from the resin of *Pinus maritima*. Fractional crystallisation from acetone yielded a solid, m.p. 132—134°, $[\alpha]_D$ —112.20° to —111.08°, which on keeping became yellow, and the rotation changed slowly, in benzene from —41.69° to +12.9° in 365 days, and in alcohol from —60.47° to +4.5° in 430 days, whilst the m.p. fell to 90°. This was due to atmospheric oxidation. The solid (colophony) obtained by distilling the liquid resin had $[\alpha]_D$ +31.65° to —21.82° in benzene and 0° to —74.79° in alcohol, depending on the conditions of distillation and dispersion.

M. ZVEGINTZOV.

Testing of sealing wax. R. NITSCHÉ (Farben.-Ztg., 1928, 33, 2794—2796).—Sealing waxes are graded by examination of their combustibility, degree of non-adhesion to the seal, adhesion to paper, and "flow" and "drop" points (Ubbelohde). Standard methods of testing and specifications for three grades are detailed.

S. S. WOOLF.

Survey of modern synthetic resins. A. A. DRUMMOND (Trans. Inst. Rubber Ind., 1928, 4, 40—58).

Sulphate-turpentine. POSTOVSKI and PLJUSNIN.
Cellulose ester solvents. WOLFF.—See V.

PATENTS.

Lacquering of metal surfaces. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of L. E. BARRINGER (B.P. 283,162, 5.1.28. U.S., 5.1.27).—A glycerol-phthalic acid type resin is applied by the usual processes to a metal surface as a primer for subsequent cellulose lacquer coatings.

S. S. WOOLF.

Preparation of [resinous] condensation products. I. G. FARBENIND. A.-G., Assees. of K. SCHIRMACHER and A. VOSS (G.P. 446,999, 16.12.21).—Neutral resins, soluble in linseed oil, are obtained by the action of formaldehyde etc. in presence of zinc chloride etc. if desired, on the condensation products of benzyl chlorides with aromatic hydrocarbons or their derivatives. E.g., the oily product from benzyl chloride and xylene is boiled with paraformaldehyde and acetic acid in presence of zinc chloride to give a highly viscous oil suitable as a plasticiser for films. From naphthalene and xylyl chloride and formaldehyde, a resin giving light-fast lacquers is obtained. Other formaldehyde condensations described are those with benzylphenols (for a copal substitute in light-fast oil varnishes), with

"tricresol"—chlorobenzyl chloride (for a thermo-hardening resin), with salicylic acid-benzyl chloride (for a shellac substitute), and with benzyl- β -naphthol (resin for light-fast lacquers). C. HOLLINS.

Production of condensation products from waste and wash-liquors and distillates of phenol-formaldehyde resin manufacture. E. GLASER (Austr.P. 106,018, 27.5.21).—The waste liquors or distillates are treated below 60° with acids or acid salts to precipitate (after some days) a resin suitable for lacquers. C. HOLLINS.

Resinous composition. V. H. TURKINGTON, Assr. to BAKELITE CORP. (U.S.P. 1,677,417, 17.7.28. Appl., 7.7.27).—A non-phenolic resin is added to a phenol-fatty oil-methylene reaction product, whereby the separation of insoluble compounds is prevented. F. G. CLARKE.

Manufacture of coloured masses or solutions thereof. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 293,067, 24.2.27).—Organic dyes, containing sulphonic or carboxylic groups, in the form of their amine salts (other than guanidine salts), are incorporated with cellulose esters or ethers, synthetic or natural resins, etc., or varnishes containing these. Examples of suitable amines are mono- and di-cyclohexylamines, piperidine, benzylaniline, Auramine base, Victoria Blue base, Rhodamine B base, Safranin base. C. HOLLINS.

Printing colours or inks, and hectograph compositions, inking rollers, etc. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 292,655 and 292,894, 16.3.27).—Sorbitol, or one of its derivatives obtained by the action of heat or alkylating or acylating agents, is added (A) as a solubilising agent to printing pastes (including vat dye printing pastes), printing inks, hectograph inks, etc., (B) as a thickening agent to carriers for such printing pastes and inks, e.g., hectograph compositions, inking rollers, blanketing cloth, etc. For both purposes sorbitol acetate is particularly useful. C. HOLLINS.

Manufacture of printing inks. J. AZZOPARDI (B.P. 293,238, 29.9.27).—Printing inks of good consistency and adhesiveness and having sufficient penetration, drying capacity, etc. are produced from straight steam-still asphalt, a volatile mineral oil, and a pigment such as lampblack, with or without the addition of turpentine, rosin, and resinates. C. O. HARVEY.

Condensation product of urea and formaldehyde. A. GAMS and G. WIDMER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,676,543, 10.7.28. Appl., 20.5.26. Switz., 5.6.25).—See B.P. 253,094; B., 1926, 756.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Changes in rubber on ageing. M. SAGAJILO (Przemysl Chem., 1928, 12, 184—190).—A sample of aged rubber contained, in 1927, 22.8% of acetone-soluble substances as compared with 2.26% in 1925. The residue after extraction with acetone contained 6.0% of chloroform-soluble substances as against 2% originally, and the content of substances hydrolysed by alcoholic caustic soda in the residue after chloroform extraction had risen from 2 to 10%. R. TRUSZKOWSKI.

Hydrogen-ion concentration [in rubber sera]. L. R. VAN DILLEN (Arch. Rubbercultuur, 1928, 12, 144—146; Med. Proefstat. Rubber Buitenzorg, 1928, No. 30, 84—86).—Experiments on the coagulation of latex with sodium fluosilicate indicate that there is a relation between the clarity of the serum and the p_H of the solution. A good coagulation without loss of rubber is obtained only between p_H 4.5 and 4.4. This may be obtained by suitable dilution of the latex. A. R. POWELL.

Colouring of cold-cured rubber. W. E. SANDERSON (Trans. Inst. Rubber Ind., 1928, 4, 22—39).—See B., 1928, 493.

PATENTS.

Manufacture of vulcanised rubber [accelerators]. W. P. TER HORST, Assr. to RUBBER SERVICE LABORATORIES CO. (U.S.P. 1,672,548, 5.6.28. Appl., 6.5.27).—Tetrasubstituted guanidines such as can be made by interaction of cyanogen chloride with secondary bases (piperidine, ethyl-*o*-toluidine) are used as vulcanisation accelerators. C. HOLLINS.

Vulcanisation of rubber. R. V. HEUSER, Assr. to A. C. BURRAGE (U.S.P. 1,677,235, 17.7.28. Appl., 5.6.28).—Rubber is heated with a vulcanising agent and a substituted guanidine containing an aryl group in each of the amino-groups and a *p*-substituted aryl group in the imino-group. B. FULLMAN.

Manufacture of rubber and compounds thereof. DUNLOP RUBBER CO., LTD., and D. F. TWISS (B.P. 293,502, 19.4.27).—Colophony and viscous liquid or adhesive semi-solid rubber accelerators and/or anti-agers and/or softening agents (e.g., aldehyde-anilines, oleic acid, etc.) are mixed in the molten or dissolved condition. The product, flakes or fragments of m.p. or softening point 80—150°, is incorporated with rubber. B. FULLMAN.

Polymerised vinyl alcohol etc. (U.S.P. 1,672,156).—See III.

XV.—LEATHER; GLUE.

Chemistry of liming [hides]. G. D. McLAUGHLIN, J. H. HIGHBERGER, and E. K. MOORE (J. Amer. Leather Chem. Assoc., 1928, 23, 318—336).—Amines are formed prior to liming and small quantities are formed during the liming process. A number of commercial lime liquors contain small quantities of volatile amine, which have an effect on the unhairing. The presence of salt inhibited the action of the amine. Diamines, e.g., ethylenediamine, possess greater unhairing power than methylamine, but their action occurs in the initial stage of liming. Secondary and tertiary amines were not detected in commercial mellow limes. An increase in the concentration of primary amines in a lime liquor diminished the time of unhairing. Preliminary swelling in pure lime did not reduce the time of unhairing in a lime-amine liquor when skin was immersed in a lime-amine solution, and equilibrium was reached after 24 hrs. between the amine, lime solution, and the skin; 20% of the original amine present was found in the skin and 80% in the liquor. Lime was absorbed more rapidly in the presence of amine, probably due to the activation of the protein carboxyl group by the amine. D. WOODROFFE.

Re-use of salt in the curing of animal skins. G. D. McLAUGHLIN, I. H. BLANK, and G. E. ROCKWELL (J. Amer. Leather Chem. Assoc., 1928, 23, 300—318).—The re-use of salt in curing causes a 1—3% increase in the initial inoculation, and introduces organisms which are more resistant to salt and show proteolytic activity in presence of a higher percentage of salt.

D. WOODROFFE.

Miscellaneous tannery investigations. G. GRASSER (J. Coll. Agric. Hokkaido Imp. Univ., 1928, 20, 219—232).

—**I. Soluble methylene compounds of tannin anhydrides.** It is concluded that 2 mols. of tannin condense when heated, forming insoluble phlobaphen, which with formaldehyde yields soluble methylenephlobaphen; the latter with dilute sodium hydroxide solution forms a sodium derivative not precipitable by acid or gelatin, but with concentrated sodium hydroxide solution forms a sodium compound of phlobaphen, from which the original phlobaphen is liberated on acidification.

II. Methylene derivatives of tannin. Solubility relationships are recorded.

III. Phloroglucinol reaction of tannin. The extract is placed on filter paper impregnated with 2% vanillin solution and acidified with concentrated hydrochloric acid; in presence of phloroglucinol a cherry-red colour develops.

IV. Behaviour of the albumin-globulin and of the coriine of the skin towards protein reagents. Positive reactions with albumin-globulin (1), coriine (2), and gelatin (3) were obtained as follows:—biuret 1, 2, 3; Molisch 3; sulphur reaction 1, 2; phenol precipitation 2, 3; mercuric chloride 1, 2, 3; tannin 1, 2, 3; potassium mercuric iodide 1; picric acid 2, 3.

V. Rate of conversion of green into violet chromic salts. Addition of sodium acetate or oxalate appears (visually) to promote the conversion of green into violet chromic sulphate solutions.

CHEMICAL ABSTRACTS.

Combination chrome and vegetable tanning. G. GRASSER and HIROSE (J. Coll. Agric. Hokkaido Imp. Univ., 1928, 20, 203—217).—In leather tanned 1 day with a mixture of basic chromic sulphate and oak-gall tannin, contraction on boiling increased with increasing proportion of the latter; in leather tanned 8 days the contraction for chromium:tannin $\geq 1:1$ was the same as for chrome leather. Leathers tanned 1 day with the ratio 7:3 to 3.5:6.5 showed minimum acid swelling, but leathers tanned 8 days in liquors containing any chromium showed the slight swelling characteristic of chrome leather.

CHEMICAL ABSTRACTS.

Colour-producing constituents of cacao bean. ADAM.—See XIX.

PATENTS.

Manufacture of tanning materials from sulphite-cellulose waste liquors. E. J. ROSENZWEIG (F.P. 620,394, 16.7.26).—Sulphite-cellulose waste liquors free from iron are heated with alkali carbonates or hydroxides for a short time at not too high a temperature. The product may be acidified with sulphuric, hydrochloric, or acetic acid, or mixed with a synthetic tannin.

D. WOODROFFE.

Manufacture of synthetic tannins. SOC. CHIM. POUR L'IND. DU CUIR (F.P. 573,416—7, 1.2.23).—Aromatic sulphonic acids are condensed with (A) 15—40% of their weight of acetone, mesityl oxide, or phorone, or (B) 20—40% of their weight of lactic acid in presence of a dehydrating agent, e.g., 1—2% of cupric or mercuric sulphate. The products in each case are neutralised with an aliphatic or aromatic base. D. WOODROFFE.

Dyeing leather. A. J. CLERMONT (F.P. 614,460, 13.4.25).—Leather with a very greasy surface is treated with a lukewarm solution of soap, rinsed, mordanted with ammonia or soda, dyed, dried, staked, and finished as usual. Vegetable-tanned leather is preferably degreased with a fat solvent. D. WOODROFFE.

Rendering leather soft and pliable. E. DESFARMET and F. SCHMITT, Assees. of ASSOC. PARISIENNE POUR L'IND. CHIM. (F.P. 615,951, 1.10.25).—Neutral or acid esters of the higher fatty acids with aliphatic, aromatic, or terpene alcohols, alone or in mixtures, or dissolved in suitable solvents or with additions of substances which increase the absorption of the ester by the hide fibres, or of soluble dyestuffs or pigments, are used on leather instead of glycerin or castor oil. The most effective products are benzyl ricinoleate or butyl oleate in acetone solution. D. WOODROFFE.

Waterproofing leather and composition therefor. E. D. VAN TASSEL, and VAN TASSEL Co. (B.P. 293,062, 31.1.27).—Paraffin wax is heated to 153° or higher with 5—30% of rubber-like gum, and the leather is immersed in the mixture at 90—100°.

D. WOODROFFE.

Glueing process and preparation of glue therefor. C. KLINGSPO (B.P. 293,232, 9.9.27).—A fluid condensate of phenol and formaldehyde is added at about 75° to a solution of glue in water or to the water in which glue is steeped. The glued articles are gradually heated to a temperature not above 200°, advantageously 145°, during 10—36 hrs.

D. WOODROFFE.

Leather cement. I. G. FARBENIND. A.-G., Assees. of F. FUNCKE (G.P. 444,387, 4.8.25).—Methylene chloride is added to an acetone solution of nitrocellulose containing a plasticiser.

D. WOODROFFE.

Fibres from skins of fish (B.P. 284,297).—See V.

XVI.—AGRICULTURE.

Influence of potash manuring with increasing nitrogen dressings on the yield and quality of barley in 1927. H. WIESSMANN and E. SCHRAMM (Z. Pflanz. Düng., 1928, 7B, 314—322).—Potash manuring alone does not increase the protein content of barley, but a small increase results from an additional fairly heavy dressing of nitrogen. The "weight per thousand grains" was not affected by potash salts, but the conjoint use of nitrate tended slightly to increase the proportion of small grains at the expense of the large ones.

A. G. POLLARD.

Comparison of the Neubauer seedling method and the Lemmermann citrate method [for determining the phosphate requirement of soils] with field trials at Aschersleben. G. SCHULZ (Z. Pflanz. Düng., 1928, 7B, 297—301).—Extensive comparative experiments are recorded. Good general agreement is

shown among the three methods for cereals, but results diverge considerably with sugar beet. By increasing the limiting figure for phosphate in the Lemmermann process from 20 to 25 mg. slightly better agreement is shown.

A. G. POLLARD.

Nitrogen fixation by micro-organisms. A. RIPPEL and H. POSCHENRIEDER (J. Landw., 1928, 76, 101—112).—Examination of the energy relationships of nitrogen fixation in the nodules of legumes shows the process to be less economic than was hitherto supposed if the exothermic formation of ammonia from its elements is considered. Alternatively, since the necessary hydrogen can, in the circumstances, arise only from organic decomposition, the nitrogen fixation process may be considered from this point of view.

A. G. POLLARD.

Tapioca. I. Sampling the standing crop. II. Coeruleomolybdate method for determination of phosphates. V. R. GREENSTREET (Malayan Agric. J., 1928, 16, 59—69, 70—75).—II. The effect of certain constituents of the soil on the accuracy of the method is described. In silica-free solutions of tapioca tuber and oil-palm leaflet, the results obtained are in satisfactory agreement with those given by the gravimetric method, but with tapioca leaf and stem, pineapple ash, and soil, divergences of as much as 31% may occur. The use of the strongly acid "Kjeldahl" solution without removal of silica invariably leads to low results.

F. R. ENNOS.

Relative solubility of phosphoric acid and potassium in German and tropical soils. H. VAGELER (Z. Pflanz. Düng., 1928, 11A, 89—93).—Determinations of phosphoric acid and potassium soluble in 10% hydrochloric acid and taken up by plants in the Neubauer method were made on series of German and Rio Magdalena soils. In the case of the German soils there was no parallel between the acid-soluble and Neubauer potash, but the two methods gave comparable results in a number of the tropical soils. In contrast to the potash, tropical soils were much lower in acid-soluble phosphate than the German soils.

H. J. G. HINES.

Solubility of rock phosphates as affected by plant roots. M. DOMONTOVITSCH and A. SCHESTAKOV (Z. Pflanz. Düng., 1928, 11A, 108—112).—Oats and millet grown in sand cultures supplied with phosphorite as a source of phosphoric acid made little growth. When buckwheat and lupins were grown in the same pots the growth of the cereals was much improved, particularly in the case of lupins. At the same time the p_H value of the soil solution was increased slightly by both buckwheat and lupins, resulting in increased solubility of the phosphate.

H. J. G. HINES.

Uptake and utilisation of plant nutrients. W. U. BEHRENS (Z. Pflanz. Düng., 1928, 11A, 93—107).—Vegetation experiments with oats were made in sand cultures. The effects of supplying nutrients by continuously percolating solutions were compared with those obtained by supplying full amounts of nutrient initially. It was shown that the uptake from solutions containing only 2.2 mg./litre of nitrogen and 0.65 mg./litre of phosphoric acid was as good as from stronger solutions. In discussing the evaluation of insoluble phosphates in

the light of these experiments, stress is laid on the period at which the nutrients become available and are taken up by the plant.

H. J. G. HINES.

Mathematical expression of the action of nutrients. W. U. BEHRENS (Z. Pflanz. Düng., 1928, 11A, 150—155).—The Mitscherlich equation is slightly modified and extended.

H. J. G. HINES.

Effects of manurial treatments on the chemical composition of gooseberry bushes. I. Effects on dry matter, ash, and ash constituents of leaves and stems of terminal shoots and of fruits, and on total nitrogen of fruits. T. WALLACE (J. Pomology, 1928, 7, 130—145).—A deficiency in soil potash influenced the growth and composition of gooseberry bushes, and is characterised by (a) high dry matter content of the leaves and stems of terminal shoots, (b) low ash content in the dry matter of stems and berries, and (c) high proportion of calcium, magnesium, sodium, and phosphorus in the ash of stems, leaves, and berries. Variations in the lime content of the ash resulting from a potash deficiency are greatest in the leaves and smallest in the berries. The reverse is the case with the phosphate content of the ash. The effect of a deficiency of a second nutrient element on the ash analysis is masked by a deficiency of potash.

A. G. POLLARD.

"Spontaneous" ignition in stable manure. L. H. JAMES, G. L. BIDWELL, and R. S. MCKINNEY (J. Agric. Res., 1928, 36, 481—485).—A case of "spontaneous" combustion of a pile of heating stable manure is reported. Oxygen aeration of a small section of the heating material produced a rise of temperature of 26.5° in 30 min., thus affording evidence in agreement with the theory that spontaneous ignition is the result of rapid oxidation.

E. A. LUNT.

Toxic value of fluosilicates and arsenicals as tested on the plum curculio. O. I. SNAPP (J. Econ. Entomol., 1928, 21, 175—178).—The order of diminishing toxicity is: sodium fluosilicate; arsenates; lead, barium, calcium, zinc, magnesium, manganese, aluminium; sodium fluosilicate and lime; scorodite. Sodium fluosilicate is toxic to peach foliage and fruit.

CHEMICAL ABSTRACTS.

Making mechanical analyses of soils in fifteen minutes. G. J. BOUYOUCOS (Soil Sci., 1928, 25, 473—480).—If the particles of the soil are grouped into three main groups, viz., combined sand, silt, and clay or colloids, these groups can be determined in 15 min. by the hydrometer method.

H. J. G. HINES.

Pentosan content in relation to winter hardiness in the apple. W. A. DELONG (Sci. Agric., 1928, 8, 501—523).—In general, the least hardy varieties of apples have the greatest pentosan content. The pentosan content is not correlated with the total loss or rate of loss of moisture at 75° in either aerial or subterranean portions of the tree. The discrepancy between results obtained by the hydrochloric acid-phloroglucinol and fermentation-copper reduction methods is not due to substances precipitable by phloroglucinol and soluble in 95% ethyl alcohol at 60°. The small fraction of the phloroglucide precipitate soluble in alcohol is probably derived from hexoses.

CHEMICAL ABSTRACTS.

Saidel's method for the investigation of aqueous soil solutions. E. ANGELESCU (Bul. Soc. chim. România, 1928, 10, 14—25; cf. Saidel, IV^{me} Conf. de Pedologie, Rome, 1925, 2, 556).—Polemical against Saidel, whose method differs from that of Wolf (Landw. Jahrb., 1873, 2, 391) only in the manner of calculation, which takes no account of substances remaining from a previous extraction. C. HOLLINS.

Sulphur content of Alberta soils. F. A. WYATT and J. L. DOUGHTY (Sci. Agric., 1928, 8, 549—555).—In semi-arid soils the sulphur content is often greater than the phosphorus content, and increases with depth. Concentration zones of soluble salts are formed 3—6 ft. below the surface; the zone of sulphate concentration is usually below the lime layer. The result of leaching is shown better by the depth of the zone of calcium carbonate accumulation than by that of sulphate concentration. Alberta alkali soils are described.

CHEMICAL ABSTRACTS.

Determination of nitrogen in fertilisers. THOREL (Ann. Falsif., 1928, 21, 257—261).—For fertilisers containing nitric nitrogen, the aqueous extract of 4 g. is diluted to 200 c.c., and nitric nitrogen determined on 20 c.c. by Schloesing's method. Potassium is determined in another 25 c.c., and in the absence of urea or its soluble compounds ammoniacal nitrogen is determined on 50 c.c. by means of magnesium. The insoluble residue from the extract is submitted to the Kjeldahl treatment, and the ammoniacal nitrogen present found by distillation in the Schloesing-Aubin apparatus. The organic nitrogen also is thus obtained. Total nitrogen is determined on 1 g. by a Kjeldahl distillation, with a preliminary destruction of nitric nitrogen by means of ferrous chloride and hydrochloric acid, and the organic nitrogen deducted from this gives the ammoniacal nitrogen. If nitric nitrogen is absent, the same procedure is followed, omitting the treatment with ferrous chloride. D. G. HEWER.

Determination of nicotine in sprays. R. SCHERPE (Zentr. Bakt. Par., 1927, II, 71, 93—105).—The fluid (6 c.c.) is mixed with saturated lead acetate solution (0.6 c.c.) and powdered sodium sulphate (0.32 g.), the mixture filtered into a graduated cylinder, and the residue washed with 1% sodium sulphate solution. Silicotungstic acid (15% solution, 3 c.c.) is added, the liquid diluted to 20 c.c., and kept for 24 hrs.; any surface precipitate is shaken down and the height of the column h is read after 2 hrs. If $h > 2$ cm., $h \times 0.70 =$ mg. of nicotine; if $h < 2$ cm., $h \times 0.83 =$ mg. of nicotine. CHEMICAL ABSTRACTS.

Soils of Bowie, Denton, Freestone, and Red River Counties. G. S. FRAPS (Texas Agric. Exp. Sta. Bull., 1927, No. 375, 48 pp.).

PATENTS.

Producing soluble phosphate fertilisers. F. G. LILJENROTH (B.P. 282,619, 20.4.27. Sweden, 23.12.26).—Crude calcium phosphate is leached with a solution containing sulphuric acid and ammonium sulphate, the latter obtained from the precipitated calcium sulphate by treatment with ammonia and carbon dioxide. The ratio of acid to sulphate in the solution is 7 : 2 by weight.

The product is an ammonium phosphate possibly containing some sulphate. B. FULLMAN.

Manufacture of insecticides for spraying plants and pickling seeds. FARBENFABR. VORM. F. BAYER & Co. (F.B. 610,919, 27.11.25. Ger., 3.2.25).—Mercuriated organic compounds are treated with alkali in the form of alkali phenoxides, borax, etc. to give soluble insecticides which are applied in solution with salt, ferrocyanide, or other addenda. Suitable mercuriated compounds are anhydronitro(hydroxymercuri)-phenol or -cresols, chloro(sulphatomercuri)phenol, aceto-mercuriphenol, cyanomercuricresol, anhydrohydroxymercuriphenol, mercuribenzoic acid, chloro(mercuri)-naphthols, etc. C. HOLLINS.

Eradication of plant pests. I. G. FARBENIND. A.-G. (F.P. 615,634, 5.5.26. Ger., 15.5.25).—Aqueous ammonia, alone or in conjunction with other parasite-destroying agents, is used in such amount as will combine with the humic acid of the soil and thus lose its injurious action on the plants themselves. The quantity recommended is 1000—1500 kg. of 0.5—1.0% aqueous ammonia for each hectare of soil infested with parasites. C. HOLLINS.

[Apparatus for] drying crops. J. CAMPBELL (B.P. 294,383, 3.8.27).

XVII.—SUGARS; STARCHES; GUMS.

Working white sugar massecuites [in the beet sugar factory]. F. NOSEK (Z. Zuckerind. Czechoslov., 1928, 52, 457—461).—Microscopical observation of the centrifuging of granulated massecuites shows the mother-liquor frequently to contain such an amount of extremely fine grain as to impart to it a milky appearance. On turning on cold or particularly hot water for covering, after the adhering syrup has been washed out, the crystal begins to be attacked. Steam has a similar effect, though less severe than with hot water, but it also causes the formation of fissures in the wall of sugar in the centrifugal through which the condensed water penetrates, causing further etching of the crystal. J. P. OGILVIE.

Chemical theory of [sugar] saturation. I. O. SHNAIDMAN (Nauch. Zapiski, 1928, 5, 273—278).—The alkalinity and polarisation of a solution of sugar precipitated from a sugar-lime solution by carbon dioxide, and treated with a current of air free from carbon dioxide, depend on the time elapsing before analysis. In the first stage of saturation a colloidal solution of lime forms; with decrease in alkalinity this begins to gelatinise. The amorphous calcium carbonate adsorbs both sucrose and the calcium oxide (1 : 1) compound; as the amorphous substance crystallises the absorption decreases. CHEMICAL ABSTRACTS.

Crystallisation of second-product [sugar] fill-mass. B. E. KRASILSCHIKOV (Nauch. Zapiski, 1928, 5, 309—312).—With a fill-mass of 93.63—93.17° Brix, crystallisation progressed with cooling; only below 45° did the coefficient of supersaturation slightly increase. With a fill-mass of 94.55—94.09° Brix, and with more rapid cooling, the coefficient of supersaturation immediately increased and remained high.

CHEMICAL ABSTRACTS.

Washing the filter-press mud [in sugar manufacture]. B. E. KRASILSCHIKOV (Nauch. Zapiski, 1928, 5, 287—290).—Cold water removes more sugar than ammoniacal hot water, which removes a greater proportion of solids. CHEMICAL ABSTRACTS.

Undetermined sugar losses in the process of defeco-saturation. L. O. SHNAIDMAN (Nauch. Zapiski, 1928, 5, 322—328).—The loss of sugar is caused by the absorptive power of the lime; its magnitude depends on the amount of lime precipitated in the saturation process, the lime added in the defecation process, the concentration of sucrose, the temperature, and the reaction of the saturated juices. The absorbed substances normally contain sugar representing a loss of 0.7—0.9%. CHEMICAL ABSTRACTS.

Acids of maple syrup. E. K. NELSON (J. Amer. Chem. Soc., 1928, 50, 2006—2008).—These are present mainly as salts. They have been isolated from samples from two different localities by acidification followed by (a) ether extraction, (b) removal of volatile acids in a vacuum, and (c) precipitation as lead salts. Malic acid predominates (approx. 0.1% of syrup), with formic, acetic, and citric acids (approx. 0.01% of each) and traces of fumaric, succinic, and an unknown acid of high m.p. Tartaric and tricarballic acids were not detected (cf. von Lippmann, A., 1915, i, 362). H. E. F. NOTTON.

Acids of maple sugar "sand." E. K. NELSON (J. Amer. Chem. Soc., 1928, 50, 2028—2031).—The sandy deposit of calcium salts formed during the concentration of maple syrup contains 51% of mineral matter. In addition to malic acid, small quantities of formic, acetic, fumaric, succinic, and citric acids are present, and traces of *d*-tartaric acid, tricarballic acid, and an unknown acid (*hydrazide*, m.p. 173—175°) have been isolated. H. E. F. NOTTON.

Flavour of maple syrup. E. K. NELSON (J. Amer. Chem. Soc., 1928, 50, 2009—2012).—The ethereal extract of maple syrup from which most of the sugar has been precipitated by means of barium hydroxide contains acids, phenolic derivatives, and a *substance*, m.p. 212°, which is not identical with hydrophlorone, but yields a volatile quinone with ferric chloride. The maple flavour is destroyed by dissolving in alkali, and it cannot be recovered unchanged after adsorption on norit. Direct ether extraction of the syrup yields a product with an intense odour of maple, which arises principally from unstable resinous phenolic substances. The extract also contains a *product*, m.p. 210—212°, which does not yield a quinone on oxidation, and *aldehydes*, one of which, m.p. 74—76°, resembles but is not identical with vanillin or ethylvanillin. It is oxidised by ferric chloride to a substance resembling dehydrodivanillin. H. E. F. NOTTON.

Manufacture of maize starch and the extraction of the residues and maize oil. O. K. A. KRIZKOVSKY (Chem.-Ztg., 1928, 52, 425—428, 466—467, 486—487, 526—528).—A detailed description of the various stages in the process of manufacture employed in Germany. 100 pts. of maize containing 13 pts. of water and 5 pts. of water-soluble matter yield on the average 52.33 pts. of starch, 22.77 pts. of fodder, 3.87 pts. of oil

cake, and 2.5 pts. of maize oil, all calculated on the dry basis. F. R. ENNOS.

Chloroform test in evaluation of the quality of starch. W. PLAHL (Z. Unters. Lebensm., 1928, 55, 295—296).—The abnormal colour of starch of inferior quality becomes much more evident when the sample is moistened with chloroform. W. J. BOYD.

Honey. ELSER.—See XIX.

PATENT.

Extraction of sugar from beet. R. G. W. FARNELL (B.P. 293,066, 23.2.27).—Beet cosettes are dried and extracted with water at a temperature not exceeding 50—60° and under reduced pressure, which may be varied. F. R. ENNOS.

XVIII.—FERMENTATION INDUSTRIES.

Autolysis of bottom-fermentation yeast. W. WINDISCH, P. KOLBACH, and E. F. ROTHENBACH (Woch. Brau., 1928, 45, 251—256, 261—265, 281—285, 298—302).—The autolysis was carried out on 30-g. portions of washed yeast, freed from hop resins by treatment with 10% sodium carbonate solution, in 250 c.c. volume and adjusted to the required reaction by addition of 0.1N-hydrochloric acid and caustic soda. By working at 47°, autofermentation and bacterial action were suppressed. Total solids and "formol" and total nitrogen were determined on the filtrates from the boiled digestions. The controls were stirred for 15 min. with 250 c.c. of water at 15° in place of the digestion. Fresh yeast must be employed for this work, as changes occur in a few days even at 2°. Autolysis proceeds most rapidly at p_H 5, the optimum for yeast pepsinase, and for 3-hr. digestions the most favourable temperature is 55°, for 6-hr. digestions 52°, the latter conditions giving 89% of the total nitrogen in solution. The proteins are still further broken down if after a time the reaction is readjusted to p_H 6.25, apparently owing to the action of a tryptase or pepsidase. The autolysis of yeast plasmolysed by chloroform proceeds most rapidly at about p_H 6. F. E. DAY.

Fermentation carbon dioxide. F. STOCKHAUSEN and F. WINDISCH (Woch. Brau., 1928, 45, 277—281, 289—298, 305—311, 317—324, 329—333).—Fermentations carried out in flasks in which the carbon dioxide was retained under pressures of 0.0066—1.0 atm. are compared with similar fermentations in flasks plugged with cotton wool. In all cases those under pressure show a slight retardation of the rate of fermentation, though the same final values for p_H and attenuation are reached and clarification is improved. The yeast, though less in quantity, is of better microscopical appearance and fermentative activity. Similar results are obtained at all the pressures employed and at 0.0066 atm. applied from the end of the first to the end of the fifth day. Since also yeast increase, p_H , and attenuation reach the same final values at 2.5°, 10°, and 20°, it is concluded that the difference between open and closed fermentation is mainly due to more complete exclusion of air from the latter. A series of four successive fermentations under 0.0066 atm. pressure indicates that under such conditions yeast maintains its

vitality better than in open fermentations. The possible industrial uses of carbonic acid are discussed.

F. E. DAY.

Colorimeter [for wort etc.]. K. S. FELIX, HELMRICH, and WANDERSCHECK (Woch. Brau., 1928, 45, 312—315).—A parallel beam of light from an incandescence lamp is passed down a vertical cylinder carrying a window at the lower end. The cylinder can be lowered by rack and pinion into a glass-bottomed vessel containing the liquid to be tested. The ray then passes through a yellow filter, to decrease sensitivity to the violet end of the spectrum, and finally impinges on a potassium photo-electric cell. The current passing through this is read on a milliammeter. The zero of the instrument is obtained by bringing the movable cylinder down till the window is in contact with the bottom of the containing vessel and inserting a removable comparison slide of suitable colour. The ammeter reading is taken, the slide removed, and the cylinder raised till the depth of liquid in the container is sufficient to bring back the ammeter reading to the original figure. This depth is measured on a scale attached to the cylinder. The method of calibrating the instrument is described. The use of the yellow filter and of the principle of substitution of colour against a standard slide have overcome the original difficulty of the inconstancy of the potassium cell after a period of rest. F. E. DAY.

Stabilisers for wines. [Determination of volatile acids.] FONZES-DIAON (Ann. Falsif., 1928, 21, 266—272).—Salicylic acid (0.5 g. per litre) is recommended to be added to the sample of wine as a preservative in preference to sodium benzoate. Volatile acids in such wines may be determined by the method of Blarez, which involves direct titration in a colourless solution, but a correction of 0.1 g. should be made for combined salicylic acid. The Roos-Mestrezat method gives slightly higher results (0.05) and a correction of 0.15 g., representing 0.1 for salicylic acid and 0.05 as the mean difference for the two methods, should be applied. In Duclaux's method the volatile acidity is always low, and the French official method, although not influenced by the stabilisers, gives much too low results.

D. G. HEWER.

Spectrochemical detection of fruit wine in wine. O. E. KALBERER (Z. Unters. Lebensm., 1928, 55, 214—230; cf. B., 1927, 686).—The absorption spectra of various red and white wines with and without addition of fruit wine were examined with reference to the characteristics of the extinction curves. When 15% or more of fruit wine was present, it was detected by the shape of the extinction curve. The absorption spectrum is little affected by the presence of sulphur dioxide, but it is considerably affected by decolorisation with charcoal.

W. J. BOYD.

Detection of isopropyl alcohol in still wine by means of piperonal. G. REIF (Z. Unters. Lebensm., 1928, 55, 204—214).—The alcohol is distilled from 10 c.c. of the sample on the boiling water-bath and the distillate diluted to 30 vol.-%, 0.3 c.c. of the diluted distillate is mixed with 5 c.c. of 0.5% alcoholic piperonal solution and 1 c.c. of water, 20 c.c. of sulphuric acid warmed to 40° are added, and after 5—10 min. 3—4 c.c. of the

mixture are warmed for 3—5 min. on the boiling water-bath. A greenish-brown colour indicates the absence of isopropyl alcohol, whilst a reddish-brown to red colour shows its presence. By this method 1% of isopropyl alcohol can be detected without first destroying the aldehydes, terpenes, and acetal. Satisfactory results were also obtained using anisaldehyde instead of piperonal. The method can also be used for the detection of isobutyl alcohol. Fermentation amyl alcohol may be removed by adding 0.04 g. of animal charcoal and 2 c.c. of water to 0.3 c.c. of the diluted distillate and filtering, the filtrate being tested as described above. If 30 c.c. of 30% acetic acid are added to the reaction mixture after it has been warmed on the water-bath, in the presence of isopropyl alcohol a rose or rose-red coloration develops and persists for some time.

W. J. BOYD.

PATENTS.

Manufacture of baking yeast. H. CLAASSEN (B.P. 280,861, 26.8.27. Ger., 19.11.26).—From a mother yeast which is cultivated in a solution containing purely organic nutriment, two or more cultures are prepared in molasses solution, which contains increasing quantities of inorganic nitrogen nutriment in addition to the organic one. Increasing proportions of albumin are found in the yeasts, the first culture containing about 55%, and the last about 60%. The last culture is not compressed, but separated from the wort and used as store yeast for the manufacture of baking yeast in molasses solution alone or with the addition of other organic nitrogen nutriment.

C. RANKEN.

Production of pale, strongly hopped beers. HOFBRAUHAUS WOLTERS UND BALHORN A.-G. (B.P. 283,879, 18.1.28. Ger., 18.1.27).—The waste steam from the whole of the brewing operations is condensed and used for brewing the beer, its heat being utilised if desired. The condensate may be mixed with pre-treated deep-well water, in order to decrease the hardness of the latter prior to being used for mashing or spraying.

C. RANKEN.

Acetification of vinegar. H. FOX (B.P. 293,539, 30.5.27).—The apparatus consists of a revolving liquor receiver provided with sprayer pipes of different lengths, having perforated nozzles at the outer ends arranged to give a uniform distribution of liquor over the birch bed surface. On the discharge side of the pump a trap pipe is attached to prevent the resultant vapour and gases escaping through the pump. An atmospheric condenser on the outside of the vat acts as an outlet for the gases and vapour, returning the condensed vapour back to the vat, whilst the gases escape by a pipe to the atmosphere or may be collected.

C. RANKEN.

Production of alcohol and acetone from vegetable matter. A. C. THAYSEN, L. D. GALLOWAY, and W. E. BAKES (B.P. 293,514, 30.4.27).—Vegetable residues and tissues containing hemicelluloses or polysaccharides which liberate pentoses by hydrolysis are passed through a steeping drum containing dilute acid. The steeped material is pressed to remove surplus acid and steamed at atmospheric pressure in vats. The soluble carbohydrates are extracted by sterilised water and the

neutralised mash is fermented with pentose-fermenting types of micro-organisms, the fermented liquor being subsequently distilled to collect the volatile products.

C. RANKEN.

Manufacture of yeast. A. P. HARRISON, Assr. to FLEISCHMANN Co. (U.S.P. 1,676,437, 10.7.28. Appl., 19.10.25).—See B.P. 277,476; B., 1927, 889.

XIX.—FOODS.

Neutralisation of milk and its detection. R. STROECKER (Z. anal. Chem., 1928, 74, 1—28; cf. Tillmans and Luckenbach, B., 1925, 967).—A method depending on the fall in the electrical conductivity of milk is described. Although by the use of a mercuric chloride-hydrochloric acid serum and variously treated milk samples a better comparison of their respective acidities could be made than was possible from the corresponding Soxhlet-Henkel figures, for practical purposes it was found better to employ dialysed ferric hydroxide solution in hydrochloric acid as the serum, the fall in conductivity being found from the relation: conductivity of serum diluted with water + conductivity of the hydrochloric acid used diluted with water—conductivity of the acid-treated serum = fall in conductivity. Lactic acid alone in the acidified serum caused only negligible change in its conductivity. The method consists in mixing 50 c.c. of milk with 10 c.c. of double-distilled water and 40 c.c. of dialysed ferric hydroxide solution, the whole being vigorously stirred for some moments. After filtration, 20 c.c. of the filtrate are mixed with 30 c.c. of double-distilled water, and the specific conductivity at 18° is measured. Separate measurements are similarly made of the conductivities of a further 20 c.c. of the serum when treated with 25 c.c. of double-distilled water and 5 c.c. of 0.2N-hydrochloric acid, and of a mixture of 45 c.c. of double-distilled water with 5 c.c. of 0.2N-hydrochloric acid. The relation quoted above is then applied. Temperature control is essential. A method of constructing a practical "neutralisation curve" is explained. The conductivity method permits of the accurate determination of neutralisation over 1°, and is independent of slight variations in the milk, such as raised sodium chloride content. It is practicable for skimmed, dried, watered, or heated milk, no dilution or bringing into solution being required. The influence of the iron solution is only slight, but preservatives can considerably affect the results.

R. A. PRATT.

Apparent viscosity of milk as influenced by some physical factors. G. M. BATEMAN and P. F. SHARP (J. Agric. Res., 1928, 36, 647—674).—The viscosity coefficient of milk (whole, skimmed, or condensed skimmed) increases with the shearing force applied and approaches a constant at high shear values. Under conditions usually obtaining in processes for determining the viscosity of milk and milk products, the curvature of the viscosity-pressure curve is in the region of its greatest value, and thus small changes in pressure markedly affect the values obtained. Mechanical agitation may decrease the viscosity of whole milk, but does not affect that of skimmed or homogenised milk. The viscosity of skimmed milk increases with age, but aged skimmed

milk tends to regain its original viscosity after repeated passage through the capillary. Homogenising of whole milk increases its viscosity, but does not affect that of skimmed milk. The viscosity of skimmed milk is increased by pasteurising at 62° for 30 min. Freezing of skimmed milk decreases its viscosity, but after being maintained in a frozen condition for several days the viscosity returns to its original value. Similar effects result by alternate freezing and thawing for several days. The viscosity of skimmed milk is not a strictly linear function of the total solid content.

A. G. POLLARD.

Determination of milk in caramel etc. W. HARTMANN (Pharm. Zentr., 1928, 69, 337—339; cf. Lührig, B., 1927, 236).—The Reichert-Meissl values of a series of mixtures of butter fat and coconut oil were determined using 1 g. of the fat saponified with 4 g. of 5% glycerin-potash solution. The values obtained after correction by the divisor figure for the butter fat were considerably higher than those calculated from the Reichert-Meissl numbers of the component fats, for all mixtures up to 75% of butter fat. With increasing butter fat content above 75% the values found approached the calculated values until at 90% of butter fat they agreed. A further correction using the divisor figure 1.06 should be applied for all mixtures of butter fat and coconut oil containing less than 75% of butter fat. Still higher results are obtained if the proportion of glycerin-potash solution used for saponification is increased. It is important to maintain the exact length (52 cm.) of the condenser of the Polenske apparatus.

W. J. BOYD.

Cryoscopic examination of milk and determination of the "cryolac number" and the chlorine-sugar number. J. FIEBE and W. KORDATZKI (Z. Unters. Lebensm., 1928, 55, 251—262).—The milk of cows inoculated with various pathogenic sera showed no alteration in f.p. depression or in osmotic pressure. The f.p. depression of normal milk serum was between 0.537° and 0.576° (mean 0.552°). The "cryolac number" was between 393 and 435 (mean 413), indicating that the chloride and lactose account for approximately 75% of the total depression. The chlorine-sugar number varied from 1.63 to 6.63 for individual animals. Neither microscopical examination of the milk nor clinical examination of the animal showed any relation between high chlorine-sugar number and mastitis. The f.p. depression and "cryolac number" are shown to be useful in determining the amount of water added to milk.

W. J. BOYD.

Evaluation of milk. J. GERUM (Z. Unters. Lebensm., 1928, 55, 274—283).—The interpretation of analytical data for samples of milk is discussed. The importance of weighing all the data together, and in particular the usefulness of cryoscopic determinations in deciding whether abnormalities are due to watering or skimming of the milk or to the condition of the animal, are emphasised. The administration of aloes to the cow produces a marked but delayed lowering of the fat content of the milk.

W. J. BOYD.

Action of halogens on milk and its constituents. A. J. J. VANDE VELDE (Natuurwetensch. Tijds., 1928,

10, 66—72).—Chlorine and bleaching powder cause coagulation, the casein being richer in chlorine in the latter case. The action of bromine is similar. Successive small quantities of iodine are completely absorbed, and appear in the ash and the casein; the milk regains its white colour on keeping between each addition, and coagulation occurs only after considerable quantities have been added (1 g. of iodine powder to 100 c.c. of milk). Milk may be sterilised by suitable addition of iodine. The casein precipitated after several small additions contained 19.8% I and was stable. Chlorine and bromine oxidise lactose to mucic acid; iodine has practically no effect. S. I. LEVY.

Chemical composition of certain Italian milk-foods. L. SETTIMI (*Atti R. Accad. Lincei*, 1928, [vi], 7, 586—589).—Roman ricotta is prepared by heating the whey, obtained by coagulating milk with rennet in the manufacture of cheese, at 75—80° in presence of an organic or mineral acid. Two types exist, differing in the proportion of fat, the difference being probably due to the use of whey from whole milk in one case and of whey from de creamed milk in the other. Their mean percentage compositions are: water, 56.0, 66.3; total nitrogenous compounds, 8.51, 8.3; soluble nitrogenous compounds, 1.04, 1.1; fatty matters and phosphatides, 33.8, 23.5; lecithin, 1.16, 0.94; lactose, 0.9, 1.1; ash, 0.48, 0.49; acidity as lactic acid, 0.21, 0.23%; g.-cal. per 100 g., 353, 257. The soluble nitrogen compounds are mainly proteoses and peptones, with small proportions of amino-acids, and the ash is essentially calcium phosphate, with small amounts of chlorides and sulphates of sodium, potassium and magnesium. Two types of mozzarella are also made, the Roman from de creamed milk and those of Cardito from partly de creamed milk; their respective compositions are: water, 67.0, 61.0; total nitrogenous compounds, 27.0, 16.0; soluble nitrogen compounds, 5.1, 2.15; amino-nitrogen, 0.25, 0.12; fats and phosphatides, 3.0, 20.0; lecithin, nil, 1.05; lactose, trace, 0.5; ash, 2.42, 2.4%; g.-cal. per 100 g., 139, 253; the ash is mostly sodium chloride (47%) and calcium phosphate (30%), with small amounts of the phosphates, chlorides, and sulphates of sodium, potassium, and magnesium.

T. H. POPE.

Fat losses in buttermilk. W. B. COMBS (*World's Butter Rev.*, 1928, 2, No. 3, 9—10, 20).—The fat content of buttermilk is greatest in the spring. The Mojonier (Röse-Gottlieb) method for the determination of fat agrees with the butyl alcohol method, but the result is higher than is given by Babcock's method.

CHEMICAL ABSTRACTS.

Determination of butyric and caproic acids in butter fat. A. I. VIRTANEN [with L. PULKKI] (*Z. anal. Chem.*, 1928, 74, 321—330).—The Reichert-Meissl distillate contains 89% of the butyric acid and 95% of the caproic acid present in the butter fat. Solubility and other influences are such that the amount of caprylic acid is practically constant and equivalent to 3.5 c.c. of 0.05*N*-alkali. The amount of capric acid is negligible. To determine the amounts of butyric and caproic acids 100 c.c. of the Reichert-Meissl distillate (containing caprylic acid equivalent to 3.2 c.c.

of 0.05*N*-alkali) are distilled and 50 c.c. of distillate are collected. This distillate, which contains caprylic acid equivalent to 3.05 c.c. of 0.05*N*-alkali, and the residual liquid in the distillation flask are titrated with 0.05*N*-alkali. If *B* and *C* are the amounts of butyric and caproic acids, expressed as c.c. of 0.05*N*-alkali, in 100 c.c. of the Reichert-Meissl distillate and *x* and *y* the titrations of the distillate and residue therefrom, respectively, then $B + C = x + y - 3.2$ and $0.745B + 0.925C = x - 3.05$. These relations are valid only when the distillation occupies about $\frac{1}{2}$ hr. The amounts of butyric and caproic acids in the entire Reichert-Meissl distillate are thus calculable. The amounts of butyric and caproic acids in the butter fat itself are obtained by multiplying these figures by 100/89 and 100/95, respectively. The amount of butyric and caproic acids in 10 specimens of Finnish butter varied between 3.12% and 4.24% (mean 3.79%) and 1.38% and 2.12% (mean 1.75%), respectively. The solubilities in water at 15° of caproic, caprylic, and capric acids are 0.891, 0.072, and 0.0026 g./100 c.c. of water, respectively. J. S. CARTER.

Prolamine of wheat flour. R. HERZNER (*Z. Unters. Lebensm.*, 1928, 55, 262—274).—The proteins extracted from wheat flour by different alcohol-water mixtures are not identical. A study of the molecular binding power, specific rotations, and elementary analyses of different fractions reveals the presence of two main groups of prolamines. W. J. BOYD.

Mastication test for sand in flour. W. PLAHL (*Z. Unters. Lebensm.*, 1928, 55, 294—295).—Determinations of ash, ash insoluble in hydrochloric acid, and total mineral matter by treatment with chloroform sometimes fail to detect the presence in flour of amounts of sand sufficient to render the material uneatable. In such cases the mastication test may be of decisive importance. Fine sand, however, may not be detected by the teeth. W. J. BOYD.

Chemical composition and nutritive value of yellow and white oats. F. HONCAMP, W. SCHRAMM, and H. WIESSMANN (*J. Landw.*, 1928, 76, 113—127).—The higher feeding value of yellow oats depends on a slightly larger content of digestible protein, but more particularly on its higher starch equivalent and lower proportion of husk. A. G. POLLARD.

Colour-producing constituents of the cacao bean. W. B. ADAM (*Analyst*, 1928, 53, 369—372).—Unfermented cacao beans contain a catechu-tannin, a compound of the catechin series ("cacao catechin"), and a compound of the latter with caffeine which is resolved into its components by boiling chloroform. The colouring substances are complex alteration products of the tannin and the catechin. Cacao catechin, m.p. 229°, was obtained from unfermented Accra cacao beans by extraction with light petroleum and then with chloroform, removing fat and xanthine bases, extraction with ether under specified conditions, and purification with lead acetate. The catechin was determined colorimetrically by means of Mitchell's ferrous tartrate reagent. (Gambier catechin and acacatechin may be satisfactorily compared by this

reagent.) Cacao catechin must be used as a standard of comparison, since gambier catechin gives a bluer tint. The tannin is determined by shaking the dry ether-extracted powder with water at 60°, filtering, and adding an equal volume of a saturated solution of cinchonine sulphate. After coagulation of the precipitate the solution is filtered through a weighed alundum crucible, washed with dilute cinchonine sulphate solution, and dried. About 0.8% of catechin was found in unfermented beans, but fermentation decreases the proportion until none is present in fully fermented beans. Tannin varies from 1.7% in fermented Guayaquil beans to 2.37% in unfermented West African and 3.48% in very poorly fermented Machala beans. The average tannin content is about 1.9% on completion of fermentation.

D. G. HEWER.

Determination of hydrocyanic acid in Lima beans with special reference to the most favourable p_H for glucoside fission. S. K. HAGEN (Z. Unters. Lebensm., 1928, 55, 284—291).—The following method is recommended: 50 g. of the finely ground beans are shaken with a mixture of 244 c.c. of "citrate" and 156 c.c. of 0.1*N*-sodium hydroxide giving a p_H value of 6 (Sørensen, Compt. rend. Lab. Carlsberg, 8), and after 3 hrs. 50 c.c. of 30% tartaric acid solution are added; 250 c.c. of the liquor are distilled off in a strong current of steam into 50 c.c. of water containing 1—2 c.c. of 2*N*-sodium hydroxide. To the distillate 1 g. of potassium iodide is added and the hydrocyanic acid is determined by titration with 0.05*N*-silver nitrate (1 c.c.=2.70 mg. HCN). The experimental error of the method amounts to 0.1 mg. of hydrocyanic acid. The presence of the active glucoside-splitting ferment of the beans is indispensable in the above method.

W. J. BOYD.

Occurrence of starch in oily seed-kernels used as foodstuffs. C. GRIEBEL (Z. Unters. Lebensm., 1928, 55, 236—239).—The appearance, distribution, and detection of starch grains in sapucaja nuts (*Lecythis*), pecan nuts (*Carya olivae-formis*, Nuth.), cedar nuts (*Pinus cembra*), and pine kernels (*Pinus pinca*) are described.

W. J. BOYD.

Relationship between digestibility of fish flesh prepared by different methods and its water content. B. GLASSMAN (Z. Unters. Lebensm., 1928, 55, 231—235).—Fish flesh roasted by "dry heat" is more rapidly digested by pepsin *in vitro* than that prepared in "boiling oil" and then de-oiled, because by the former process a product of higher moisture content is obtained and the presence of the moisture moderates the polymerisation of the protein by heat. The presence of oil hinders the digestion only by mechanically preventing contact of substrate and ferment. There is no loss of protein in the "boiling oil" process.

W. J. BOYD.

Effect of onion and garlic on the growth of bacteria in sausages. E. DANIELSEN (Z. Unters. Lebensm., 1928, 55, 291—294).—The sterilising effect of onion and garlic on the growth of various aerobic and anaerobic bacteria likely to occur in sausages was found to be negligible.

W. J. BOYD.

Sanitary evaluation of flesh foods. L. M. HOROVITZ-VLASOVA (Z. Unters. Lebensm., 1928, 55, 239—246).—Determinations of alkalinity or acidity of the aqueous extract, p_H , refractive index, intensity of biuret reaction, total nitrogen, nitrogen precipitated by tannin and by phosphotungstic acid, oxidisability, and iodine value were found to be of no value as an indication of the freshness of flesh foods. The Eber reaction is useless because it is negative till after putrefaction has set in. Testing with red litmus paper for ammonia evolved on boiling with sodium hydroxide or magnesia is of no practical value because not only are ammonium salts decomposed, but ammonia is evolved from the proteins. By warming the extract or the finely minced meat in ammonia-free water after addition of magnesia for 5 min. at 50° no ammonia is removed from the proteins. Accordingly the following test is recommended: 1 g. of flesh in 10 c.c. of water and 0.1 g. of magnesia are heated at 50°, and if after 5 min. red litmus paper held above the mixture turns blue the flesh is judged to be unsound. About 0.02% of ammonia is detectable by this test. The Sørensen titration method was found useful, but the preparation of the extract introduces a source of error.

W. J. BOYD.

Cooking of foods under pressure. J. FROIDEVAUX (Ann. Falsif., 1928, 21, 252—257).—Broth prepared in an autoclave is richer in amino-acids than when made by the ordinary method, and contains more ammoniacal nitrogen than the meat from which it was prepared. The broth is poorer in nitrogenous material, in fat, and in reducing substances than ordinary broth, but the meat is better cooked under pressure and the method is more economical.

D. G. HEWER.

Determination of the inorganic constituents of honey. E. ELSE (Z. Unters. Lebensm., 1928, 55, 246—251).—The author has extended and improved the methods previously described (B., 1925, 819). The removal of ammonium salts after incineration is omitted. Phosphoric acid is precipitated as strychnine phosphomolybdate and the suspension compared with a standard preparation using a nephelometer. Iron is determined colorimetrically as the thiocyanate, manganese colorimetrically as permanganate after oxidation with potassium persulphate. Potassium is precipitated as cobaltinitrite and the washed precipitate is titrated in hot sulphuric acid solution with potassium permanganate. Calcium and chlorine are determined as described previously. Analyses are given of the inorganic constituents of four samples of Austrian honey.

W. J. BOYD.

Acids of figs. E. K. NELSON (J. Amer. Chem. Soc., 1928, 50, 2012—2013).—The following acids have been isolated: (a) from normal Adriatic figs, acetic (0.027% by weight) and citric (0.32%); (b) from Adriatic figs affected with "black neck," acetic (0.32%) and citric (0.24%); (c) from normal Calimyrna figs, acetic (0.026%), citric (0.35%), and a little malic; and (d) from Calimyrna figs affected with internal rot, acetic (0.056%), citric (0.33%), and a little malic acid.

H. E. F. NOTTON.

Chloride content of canned sauerkraut. M. E. STARK (J. Lab. Clin. Med., 1927, 12, 561—563).—

Average values for sodium chloride are 1.86 g. per 100 c.c. of juice and 1.74 g. per 100 g. of solid.

CHEMICAL ABSTRACTS.

Commercial production of sauerkraut. E. LE FEVRE (U.S. Dept. Agric., Circ. 35, 1928, 30 pp.).

Behaviour of fats in ultra-violet light. HAITINGER and others.—See XII.

Tapioca. GREENSTREET.—See XVI. **Maize starch.** KRIZKOVSKY.—See XVII.

PATENTS.

Obtaining an alimentary extract from fish. A. EHRENREICH (B.P. 284,636, 1.2.28. Belg., 1.2.27).—The flesh of the fish is heated with dilute hydrochloric acid at 70–100°, and the liquid is afterwards neutralised with sodium carbonate and concentrated *in vacuo* to a thick extract, which may be dried *in vacuo* and compressed into cubes or tablets if desired.

F. R. ENNOS.

Preservation of fruits, vegetables, etc. H. G. ZELLNER (B.P. 293,341, 4.4.27).—Fruits or vegetables with a skin or rind are scrubbed and washed in a solution made by intermixing copper sulphate and potassium permanganate with alkali hydroxide solution. After rinsing in fresh water, followed by a final washing with a solution containing sodium pentasulphide and in alkali hydroxide, the fruits or vegetables are dried.

C. RANKEN.

Production of enzymes for use in manufacture of bread. H. A. KOHMAN, K. IRVIN, and E. S. STATELER, Assrs. to FLEISCHMANN CO. (U.S.P. 1,676,446, 10.7.28. Appl., 3.10.21).—See B.P. 186,924; B., 1924, 30.

Apparatus for heat treatment of cereal substances. C. W. CHITTY, D. W. KENT-JONES, and WOODLANDS, LTD. (B.P. 294,001, 26.4.27).

Animal feed. A. B. SCHREIBER (B.P. 293,989, 27.7.27).

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Alkaloids of tobacco. A. SCHMUK (U.S.S.R. State Inst. Tobacco Invest., Bull. 44, 1928, 47 pp.).—A summary is given of the chemistry and transformations of the alkaloids of tobacco, together with a critical review of the methods suggested for the determination of nicotine in tobacco.

T. H. POPE.

Occurrence of arsenic in tobacco. H. POPP (Z. angew. Chem., 1928, 41, 838–839).—For the determination of arsenic in tobacco 200 g. are heated with fuming nitric acid until the organic matter is completely decomposed and all chlorine expelled. The solution is evaporated repeatedly with sulphuric acid to remove nitric acid, and arsenic is tested for by the mercuric chloride colorimetric method. Tobacco from the Palatinate contained 5.1, from Macedonia 0.7, from Java 0.33, and from Brazil 4.6 pts. of arsenic per million. This quantity is so small that no danger of poisoning is to be feared.

A. R. POWELL.

Determination of nicotine in tobacco and tobacco extracts. R. R. L. WORSLEY (Egypt. Min. Agric. Tech. Sci. Service Bull., 1927, 73, 5 pp.).—The following

method is suitable for use in warm climates. Nicotine sulphate solution (100 c.c.) is shaken for 10–15 min. in a well-stoppered 400 c.c. bottle with 20% sodium hydroxide solution (20–25 c.c.) and exactly 100 c.c. of light petroleum, b.p. 50–60° or above. After settling for 10 min. 40 c.c. are decanted without filtering and poured into 50 c.c. of distilled water. An excess (about 5 c.c.) of 0.1N-sulphuric acid is added, the mixture shaken gently for 1 min., and titrated with 0.01N-sodium hydroxide solution. A better end-point is frequently obtained by adding 5 c.c. of 0.01N-sulphuric acid and titrating again.

CHEMICAL ABSTRACTS.

Determination of alkaloids by the mercurimetric method. A. JONESCO-MATIU and H. VARCIVICI (Bul. Soc. Chim. România, 1928, 10, 5–8, 9–13).—I. A 1% solution of the alkaloid (1 c.c. or more) is shaken with 5 c.c. of Mayer-Valzer reagent (mercuric iodide in 10% potassium iodide solution), the precipitate is separated and washed by means of a centrifuge, and dissolved in mixed acid (1 pt. of nitric acid, 2 pts. of sulphuric acid). To the diluted solution a little permanganate is added to remove nitrous acid, then 12 drops of 10% sodium nitroprusside solution, and the mixture is titrated with 0.1N-sodium chloride until the turbidity disappears. The equivalents for atropine, aconitine, papaverine, narcotine, brucine, veratrine, and emetine are worked out from the pure alkaloids.

II. The method is applicable to atropine, quinine, strychnine, morphine in vegetable extracts such as belladonna, quinquina, nux vomica, opium, and jusquiame.

C. HOLLINS.

Apparatus for determination of alkaloids in lupins. F. KAMINSKI (Chem.-Ztg., 1928, 52, 467).—The extraction with alkaline ether-chloroform mixture is made in a bottle closed at the top with a stopper and tap, and having a side tube near the base connected through a three-way tap to a burette consisting of two 25-c.c. bulbs. On completion of the extraction, the liquid is blown over into the burette, from which aliquot portions may be drawn off for evaporation.

F. R. ENNOS.

Determination of synthetic camphor in its pharmaceutical preparations. J. BOUGAULT and (Mlle.) B. LEROY (J. Pharm. Chim., 1928, [viii], 8, 49–55).—From a study of the volatility, solubility in dilute sodium hydroxide solution, and extraction from acid and alkaline solutions of the camphoroximes the following method has been deduced: 0.5 g. of camphor dissolved in 5 c.c. of alcohol at 90° is mixed with a solution of 1 g. of hydroxylamine hydrochloride in 5 c.c. of water, and to the mixture are added 2 c.c. of sodium hydroxide solution. The liquid is boiled for 2 hrs. in a water bath, cooled, 20 c.c. of water and 3 c.c. of sodium hydroxide solution are added, and, if a precipitate or cloudiness due to camphene or borneol is formed, the liquid is filtered. The excess of alkali is exactly neutralised with hydrochloric acid and the camphoroxime is extracted with ether, 20, 10, and 10 c.c. being used in three extractions. The extracts are each washed with 5 c.c. of water, and the combined extract is allowed to evaporate spontaneously at room temperature for 12 hrs. and then dried over calcium chloride for 12 hrs. and weighed. The weight

is increased by 4% to compensate for volatility of the oxime, and the amount of camphor is $30400C/167$, where C is the corrected weight. E. H. SHARPLES.

Essential oils of Travancore. VII. Oil from rhizome of ginger, *Zingiber officinale*. K. L. MOUDGILL (J. Indian Chem. Soc., 1928, 5, 251—259).—The oil obtained in 2% yield from the scraped rhizomes of fresh, dry ginger has d_{20}^{25} 0.869, n_D^{25} 1.4891, $[\alpha]_D^{25}$ —54°, acid value 1.0, ester value 7.4. The chief constituent of the oil is zingeribene (70%), which when heated at its b.p. yields a dextrorotatory monocyclic sesquiterpene, and undergoes an analogous change on prolonged contact with air. The initial oil exhibits a similar behaviour. Old samples of ginger, green ginger, and ginger scrapings give diminished yields of oils, and the oils from the last two differ in some respects from the oil from the rhizomes. H. BURTON.

Essential oil of *Podocarpus ferrugineus*. J. R. HOSKING and W. F. SHORT (Rec. trav. chim., 1928, 47, 834—838).—The oil was steam-distilled from leaves and terminal branches of the New Zealand "miro-pine" (0.09% in autumn, 0.14% in spring). The autumn oil, d_{20}^{25} 0.9602, n_D^{25} 1.4922, $[\alpha]_{546}^{25}$ + 32.97°, contained 36% of *d*- α -pinene, 12% of cadinene, 5% of *d*-limonene and dipentene, 2% of cineole, 5% of resin, and 27% of a new diterpene, *mirene*, m.p. 102—104°, b.p. 151—152°/0.6 mm., d_{20}^{25} 0.9199, n_D^{25} 1.4890, $[\alpha]_{546}^{25}$ + 27.15° (in chloroform) (*monohydrochloride*, m.p. 97—98°). Acetic and isovaleric acids were also identified. The oil collected in spring contained much more *mirene* (47%) and less pinene (24%) and cadinene (6%). C. HOLLINS.

Determination of essential oils in drugs. R. BAUER (Pharm. Ztg., 1928, 73, 920—921).—After extracting the steam-distillate saturated with sodium chloride with pentane, the pentane is dried by filtration through filter paper and then distilled at a slightly raised temperature instead of allowing it to evaporate spontaneously. E. H. SHARPLES.

Determination of essential oils in drugs by Stamm's method. E. TIKKANEN (Pharmacia, 1926, [5]; Chem. Zentr., 1927, II, 1520).—Stamm's method has been critically examined in its application to different drugs. All determinations with one and the same powdered drug accurately agreed among themselves and gave satisfactory results. The method is inexpensive and can be carried out in 1½ hrs. E. H. SHARPLES.

Synthetic menthol. SCHIMMEL & Co. (Ber. Schimmel, 1927, 139; Chem. Zentr., 1927, II, 1519).—Synthetic menthol, m.p. 35°, produced by the authors, is indistinguishable in odour and appearance from that from peppermint oil. Its physical properties lie between those of racemic menthol produced from thymol or piperitone and those of natural *l*-menthol. E. H. SHARPLES.

Nicotine in sprays. SCHERPE.—See XVI.

PATENTS.

Production of inactive menthol (the racemate of natural menthol). RHEINISCHE KAMPFER FABR. G.M.B.H. (B.P. 285,394, 4.4.27. Ger., 15.2.27).—*i*-Menthol in the liquid mixtures resulting from the fractional

distillation of the hydrogenation products of thymol, *i*-menthone, or *isomenthone* is separated by removing *i*-neomenthol by freezing, and then fractionally distilling the residue with fresh hydrogenation product. The product of the hydrogenation of thymol or *isomenthone* below 160° contains *neoisomenthol*, m.p. 12—14°, b.p. 214.5°/760 mm. (*hydrogen phthalate*, m.p. 90—92°; *p*-nitrobenzoate, m.p. 56°). B. FULLMAN.

Manufacture of derivatives of acridine. A. G. GREEN (B.P. 293,617, 2.11.27).—Derivatives of acridines or acridinium salts, containing primary or secondary amino-groups, are converted into aldehyde-bisulphite compounds by treatment in neutral solution with bisulphite and formaldehyde, acetaldehyde, crotonaldehyde, glyoxal, etc. Examples are 3:6-diamino-acridine and acriflavine. The products are hydrolysed in the body, with production of the antiseptic acridine compounds. C. HOLLINS.

Preparation of quinoline derivatives. I. G. FARBENIND. A.-G., Assees. of R. SCHWABE, O. EISLEB, and H. JENSCH (G.P. 440,008, 29.3.24).—4-Amino-2-styrylquinolines having strong bactericidal properties are obtained by usual methods from quinaldines containing in 4-position a halogen, alkoxy-, hydrazino-, carbamyl, or carbazido-group. 4-Chloro-2-styrylquinoline, m.p. 117°, gives with alcoholic ammonia at 210—220° 4-amino-2-styrylquinoline, m.p. 173—175° (decomp.). 4-Chloro-6-ethoxy-2-styrylquinoline, m.p. 120°, similarly yields 4-amino-6-ethoxy-2-styrylquinoline, m.p. 212° (*glycollate*, m.p. 237°), also obtained by action of alcoholic ammonia on 4:6-diethoxy-2-styrylquinoline, m.p. 134° (from the chloro-compound and sodium methoxide). Reduction of 4-phenylhydrazino-2-styrylquinoline with zinc dust and acetic acid gives 4-amino-2-styrylquinoline; the 6-ethoxy-derivative is similarly prepared. 10-Chloro-8-methyl- β -naphthaquinoline, m.p. 200° (from the 10-hydroxy-compound) is condensed with benzaldehyde to the 8-styryl compound, m.p. 128°, from which 10-amino-8-styryl- β -naphthaquinoline, m.p. 226—227°, is prepared. 4-Amino-6-ethoxyquinaldine, m.p. 195°, gives with *m*-nitrobenzaldehyde 4-amino-6-ethoxy-2-*m*-nitro-styrylquinoline, which is reduced to the *m*-aminostyryl compound, m.p. 182°. 2-Styrylquinoline-4-carboxylic acid (*methyl ester*, m.p. 98°; *ethyl ester*, m.p. 77°) is converted by way of the ester into the *amide*, m.p. 274°, from which 4-amino-2-styrylquinoline is obtained by Hofmann's reaction: the *hydrazide*, m.p. 215°, yields the same product by way of the *azide* and the *urethane*, m.p. 202°. 4-Ethylamino-6-ethoxy-2-styrylquinoline, m.p. 178° [*glycollate*, m.p. 214—216° (decomp.)], and the 4-diethylamino-compound, m.p. 103°, and 4-diethylamino-6-ethoxyquinaldine, m.p. 74°, are also described. C. HOLLINS.

Manufacture of organo-arsenic compounds. R. W. E. STICKINGS, and MAY & BAKER, LTD. (B.P. 293,152, 3.5.27).—An aminoarylsarinic acid is condensed with cyanogen bromide (or other halide) to give a carbamide derivative; if a suitable substituent is present *ortho* to the amino-group, e.g., hydroxyl, carboxyl, or amino-, cyclisation takes place. Examples are: *phenylcarbamide-p*-arsinic acid from arsanilic acid, *anhydro-3-ureido-4-hydroxybenzenearsinic acid* from 3-amino-4-hydr-

oxybenzenearsinic acid, and 2:4-diketo-1:2:3:4-tetrahydroquinazoline-7-arsinic acid from 3-amino-4-carboxybenzenearsinic acid. C. HOLLINS.

Manufacture of metallo-mercapto-compounds. W. CARPMAEL. From CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 293,363, 2.2.27).—A carbohydrate containing a thiol group is treated with a salt of a metal other than silver. The *aurothiol* derivatives from 1- and 3-thioglucoses, and *bismuth* and *cadmium* salts of 1-thiogluco- glucose are described. All are readily soluble in water. C. HOLLINS.

Manufacture of hypnotic solutions. I. G. FAR- BENIND. A.-G., Assees. of L. LAUTENSCHLÄGER, M. BOCKMÜHL, and R. SCHWABE (G.P. 447,161, 29.9.25. Addn. to G.P. 446,290; B., 1928, 623).—Hypnotics of the barbituric acid or diallylacetamide type are dis- solved in water-soluble, physiologically innocuous ethers or esters of polyhydric alcohols, such as glycerol diethyl ether, or glycol monoethyl ether. C. HOLLINS.

Immunising product and method of producing the same. E. C. R. MARKS. From PARKE, DAVIS & Co. (B. P. 293,401, 31.1.27).—Antigens (known as "ecto- antigens" from a supposed association with the ecto- plasm) are obtained from micro-organisms by washing with a solvent (physiological saline etc.) for 10–15 min. B. FULLMAN.

Manufacture of elastic, easily soluble prepara- tions of pharmaceutical products, such as sticks, balls, etc. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 293,090, 25.3.27).—Sticks, balls or other shaped carriers for medicaments are made from gum tragacanth or the like rendered soluble by means of hydro- tropic substances, such as sodium toluene-*p*-sulphonate or sulphanilate. C. HOLLINS.

Manufacture of organic gold[–nucleic acid] com- pounds. A. GAMS and P. SCHEIDEGGER, Assrs. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,678,429, 24.7.28. Appl., 14.7.27. Switz., 25.8.26).—See B.P. 276,677; B., 1928, 389.

Manufacture of easily soluble anæsthetics of the *m*-aminobenzoic acid ester series. O. BILLETER, E. ROTHLIN, and J. PEYER, Assrs. to CHEM. WORKS, FORMERLY SANDOZ (U.S.P. 1,678,317, 24.7.28. Appl., 3.3.27. Ger., 10.3.26).—See B.P. 291,850; B., 1928, 623.

Preparation of salts of 3-acetyl-amino-4-hydroxy- phenylarsinic acid. E. FOURNEAU, Assr. to ÉTABL. POULENC FRÈRES (U.S.P. 1,677,964, 24.7.28. Appl., 18.1.27. Fr., 19.1.26).—See B.P. 264,797; B., 1927, 573.

***n*-Amyl[yl](valeroyl)resorcinol. *iso*Butyryl- resorcinol.** A. R. L. DOHME, Assr. to SHARP & DOHME, INC. (U.S.P. 1,677,116–7, 10.7.28. Appl., 29.4.25).—See A., 1926, 838.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Herschel effect by exposure to short wave- lengths. LÜPPO-CRAMER (Z. wiss. Phot., 1928, 25, 308– 315).—The bleaching-out of the latent image by a second

exposure to light, which up to the present has been confined to the red end of the spectrum, has now been shown to take place with yellow, green, and blue light, but only if the intensity used be very small—a con- dition which is not necessary for the longer wave- lengths. The lower intensity is required with the more penetrating short wave-lengths, so that the light may be absorbed largely in the surface of the grains where re- combination of the silver and halogen takes place, as a result of which development will be inhibited to a certain extent. The ordinary phenomenon of reversal is discussed in the light of these new facts.

J. W. GLASSETT.

Sterry effect with silver chloride. LÜPPO-CRAMER (Z. wiss. Phot., 1928, 25, 316–319).—Reagents which attack silver (e.g., 1% solutions of potassium dichromate, quinone, cupric and ferric chlorides, ammonium per- sulphate) destroy the silver chloride latent image much more readily in the higher exposed portions than in the neighbourhood of the threshold value, where the ex- posure is small. In explanation of this phenomenon it is suggested that the traces of silver first produced by light form a very stable adsorption complex with the halide, and are therefore capable of resisting the relatively weak oxidising action of the above solutions. With further exposure, the stability of the complex decreases as the amount of silver to be adsorbed increases, thus facilitating the destruction of the latent image.

J. W. GLASSETT.

Covering power of photographic silver deposits. I. S. E. SHEPPARD and A. BALLARD (Phot. J., 1928, 68, 354–366).—The change in the covering power of silver deposits by varying time of development and exposure has been studied using a *p*-aminophenol hydrochloride developer. For the two emulsions studied the covering power at all exposures decreased with time of develop- ment, but tended to increase with increased exposure. The rate of reduction of the grain is also shown to affect covering power. The following accurate method of determination of small quantities of silver has been devised. A measured area of the emulsion is treated for a short while with concentrated sulphuric acid, for partial destruction of the gelatin, and then with a few drops of 1% bromine water. The silver bromide so formed is dissolved in aqueous ammonia and converted into sulphide by the addition of sodium sulphide. The tint of this precipitate is compared with a standard in a microcolorimeter, in which a linear relation was found to exist between the colour strength and the silver content of the solution. By this method it was found possible to determine 0.0025 mg. of silver with an accuracy of $\pm 0.5\%$ and 0.01 mg. to $\pm 0.2\%$. J. W. GLASSETT.

Economic preparation of silver gelatinobromide plates, and the duration of their sensitivity. HERVÉ (Bull. Soc. franç. Phot., 1928, 15, 70–74).— Examination of emulsions made between 1881 and 1899 has led to the following conclusions: the sensitivity of silver gelatinobromide plates decreases with time, whatever the method of manufacture of the emulsion; emulsions ripened by boiling have a fine grain and are clean-working for a period of at least 40 years; fast emulsions, ammonia-ripened, have a coarser grain

and fog rapidly on keeping. The methods of making the boiled and ammonia emulsions are described.

W. CLARK.

Destruction of the image by desensitisers. LÜPPO-CRAMER (Z. wiss. Phot., 1928, 25, 282—287).—Weakening of the latent image on exposure to red light commences above a definite latent blackening so long as bromide ions are not present. Under certain conditions similar relations hold when desensitisers are used. Plates were exposed under a wedge, bathed in desensitiser, dried, given a diffuse exposure through a red filter, and finally developed. With emulsions with low free bromide content and a red filter not too deep, phenosafranine gave increased density at the low exposures and bleaching at the higher ones. Pinakryptol Green gave similar results, but to a less extent. With a dark red filter, with Pinakryptol Yellow with both medium and dark red filters, and when bromide was added to phenosafranine bleaching occurred at all exposures. The action of red light in presence of phenosafranine, and to a lesser extent Pinakryptol Green, is to give results similar to the Herschel effect under similar conditions.

W. CLARK.

Developing properties of meta-derivatives. A. and L. LUMIÈRE and A. SEYEWETZ (Bul. Soc. franç. Phot., 1928, 15, 90—92).—Homolka has shown that resorcinol has no properties as a developer, but that if methyl groups are substituted in the positions adjacent to the hydroxyl groups, the compound then acts as a developer. Pure specimens of 2:4-dimethylresorcinol, 2:4-dimethyl-*m*-phenylenediamine, and 2:4-dimethyl-*m*-aminophenol were prepared, but only the resorcinol derivative had the properties of a developer. Homolka's view that the absence of developing properties in resorcinol is due to its undergoing tautomerisation in presence of alkalis to form a diketone is questioned because it apparently cannot apply to the aminophenol and the diamine.

W. CLARK.

Photomicrography in colour. T. S. CURTIS (J. Amer. Ceram. Soc., 1928, 11, 609—632).—An outline of technique.

PATENTS.

Manufacture of photographic silver halide emulsions. O. MATTHIES, P. WULFF, W. DIETERLE, and B. WENDT, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,673,522, 12.6.28. Appl., 10.7.26. Ger., 25.7.25).—See B.P. 255,846; B., 1927, 269.

Colour photography or cinematography. L. DUFAY (U.S.P. 1,673,350, 12.6.28. Appl., 3.8.26. Fr., 4.12.25).—See B.P. 262,386; B., 1927, 461.

XXII.—EXPLOSIVES; MATCHES.

The Trauzl test. R. NEUBNER (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 1—5, 53—56, 82—87, 126—129, 162—166, 194—198).—It is proposed to modify the Trauzl lead block test by measuring the weight of explosive that is required to give on explosion an expansion of 300 c.c. This eliminates the gradual weakening resistance of the lead wall, the effect of which is a difficult factor to appraise in the usual form of the test in which fixed weights of explosive are used. As a

basis of comparison between explosives it is proposed to calculate from the weight of explosive required for an expansion of 300 c.c. the expansion that would be caused by 10 g., and to call this the coefficient of power (Kraftzahl, KZ). Directions are given for converting graphically the old Trauzl values into KZ values. The energetics of explosive reactions is discussed at some length, and expressions are obtained that enable the detonation velocities and brisance of explosives to be calculated.

S. BINNING.

Apparatus for determining ignition and explosion points of explosives. M. KOSTEVITCH (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 156—157).—An all-glass apparatus for determining the ignition point of explosives is described, which enables the first signs of ignition to be detected. For explosion point determinations a metal apparatus is used in which Wood's alloy is used as the heating agent.

S. BINNING.

Relation between temperature of explosion of a powder and its rate of combustion. H. MURAOUR (Compt. rend., 1928, 187, 289—290).—For mixtures of equal amounts of guncotton and nitroglycerin containing from 0 to 15% of centralite the logarithm of the rate of combustion is a linear function of the temperature of explosion.

J. GRANT.

Duration and length of the explosion flames of various explosives. H. KAST and H. SELLE (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 153—156).—Photographs taken on stationary and on rotating films are shown of the explosion flames of trinitrotoluene, tetryl, and kieselguhr-dynamite and several German coal-mine explosives. With trinitrotoluene three types of flame could be detected, a primary flame, a weaker secondary flame, and an after-combustion flame from the burning of detonation products in the air. No after-combustion flame was seen with the coal-mine explosives, but it is wrong to say that they give no measurable primary flame.

S. BINNING.

Liquid air as an explosive. C. BUNGE (Z. ges. Schiess- u. Sprengstoffw., 1928, 23, 44).—The view that liquid-air explosives are safer than the ordinary solid type is criticised. Even with liquid-air explosives many of the dangerous operations are still necessary, and in addition they are unexpectedly very sensitive to mechanical shock.

S. BINNING.

Mixed acid. VON BEZOLD.—See VII.

PATENT.

Nitration of cellulosic materials. A. P. H. DESBOROUGH, W. T. THOMSON, and R. S. G. KNIGHT (B.P. 293,190, 24.6.27).—Sheets of cellulosic material such as cotton are passed by a continuous band on to a roll placed above a shallow bath into which the nitrating acid is fed at the same rate as it is consumed. Only the periphery of the roll, which may consist either of spaced discs or of a perforated drum, dips below the acid.

S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Partial aeration of strong sewage with activated sludge. W. D. HATFIELD (Ind. Eng. Chem., 1928, 20, 832—835).—Experiments carried out at Decatur (Illinois)

on a sewage which consists of 60% of normal city sewage and 40% of waste from a corn-products factory confirmed results obtained at Birmingham that the volume of settled sewage which can be satisfactorily treated on percolating filters may be considerably increased by partial aeration with activated sludge prior to filtration. A pre-aeration plant is now in operation dealing with a dry-weather flow of 10 m.g./day with an aeration period of 2.5 hrs. and a settlement period of 2.6 hrs. In constructing this plant it was considered that the lower power consumption of Simplex aerators did not justify the larger initial cost of construction as compared with forced aeration through porous tiles. The excess sludge from the plant is passed into Imhoff tanks and digested with the fresh sludge. The gases produced are collected and utilised for heating the air before compression, thus reducing the power consumption in very cold weather. C. JEPSON.

Determination of stable and unstable organic matter in sewage-polluted liquids. W. E. ABBOTT (Ind. Eng. Chem., 1928, 20, 406—407).—The approximate percentage of the carbonaceous matter which undergoes change during the first-stage absorption of polluted liquids has been determined, the acid dichromate absorption being taken as a rough measure of the total organic matter. The oxygen absorbed during the first stage is usually 10% higher than that absorbed up to the beginning of nitrification, as for several days the oxidation of the last portions of the fermentable carbonaceous matter proceeds simultaneously with nitrification, but in sewages free from suspended matter the first-stage oxidation represents the oxidation of most of the soluble carbonaceous matter. The average ratio of the oxygen absorbed during the first stage to the dichromate absorption was 0.72 for raw sewage, 0.88 for raw sewage centrifuged, 0.15 for an activated sludge effluent, and 0.25 for river water. Adeney and Dawson's acid dichromate method (B., 1927, 934) and Johnson's oxy-albuminoid absorption method (B., 1927, 318) have been compared. The latter is considered unsuitable to distinguish between readily fermentable and stable carbonaceous matter. W. J. BOYD.

Titration of carbon dioxide [in water]. L. SMITH and G. WODE (Z. angew. Chem., 1928, 41, 208—212).—A comparison is made on a series of natural waters of methods for determining total, free, and combined carbon dioxide. Free carbon dioxide is titrated with 0.05*N*-caustic soda using a standard amount of phenolphthalein as indicator, matching the colour against the same amount of indicator in a buffer of p_H 8.4. For bicarbonate, titration using methyl-orange as indicator is recommended. The calculated total carbon dioxide from these determinations agrees well with that found by Westerborg's method, in which the solution is acidified with tartaric acid and distilled under reduced pressure, and the gas absorbed in baryta solution. Westerborg's method for bicarbonate (precipitation by baryta in presence of sugar and titrating the excess baryta) is inferior to the methyl-orange titration.

W. A. RICHARDSON.

Effect of certain Illinois waters on lead. O. W. REES and A. L. ELDER (J. Amer. Water Works' Assoc.,

1928, 19, 714—724).—A case of lead poisoning in this state led to an investigation of possible causes as, owing to the high mineral content of the water, such cases are rare. The amount of lead present in the water amounted to 0.08 p.p.m., which is below the amount usually considered allowable. It was found that with water containing a sodium bicarbonate alkalinity of 400 p.p.m., increasing amounts of sodium chloride produced a marked effect on the amount of erosion or lead-rusting, but had little or no effect on plumbo-solvency. Samples from various parts of the state yielded negative results as to the causes of corrosion, but indicated that a protective layer was quickly formed on the surface of the lead which practically eliminated further corrosion. C. JEPSON.

Iodine in drinking waters. J. B. ORR, W. GODDEN, and J. M. DUNDAS (J. Hyg., 1928, 27, 197—199).—Softening with lime (but not sand-filtration) decreases the iodine in water. For the determination, excess of potassium hydroxide should be added on collection, and the iodine in the water-soluble (10 c.c.) and insoluble residue determined separately.

CHEMICAL ABSTRACTS.

Analysis of insecticides. H. FRANÇOIS and L. SEGUIN (Ann. Falsif., 1928, 21, 282—286).—Methods are given for the analysis of liquid insecticides, *i.e.*, those containing formaldehyde and mercuric chloride, picric acid, nicotine and its salts, and garlic. D. G. HEWER.

Impurities of the atmosphere. A. J. H. KAM (Chem. Weekblad, 1928, 25, 298—301).—An account is given of methods of sampling and analysing factory exit gases. S. I. LEVY.

PATENTS.

Filtering apparatus. [Cleansing sand filters.] W. PATERSON (B.P. 293,534, 25.5.27).—When a sand filter is cleansed by any form of back-wash the surrounding walls have to be comparatively high to retain the sand which is expanded by the back-flow; at the end of the operation this will leave a large body of water, perhaps dirty, above the sand. In this invention this water is disposed of after the sand has settled by use of collapsible walls, a dwarf wall at one end above which is a removable flap, or merely through openings made in a wall or walls. B. M. VENABLES.

Sterilisation of water. J. MUCHKA (B.P. 290,166, 5.5.28. Austr., 7.5.27).—Water is first chlorinated (with free chlorine or chlorine compounds) and then freed from chlorine *etc.* by passage through an apparatus containing carbon or masses containing carbon. The apparatus is provided, after the manner of a sand filter, with a reflux device consisting preferably of a jet nozzle and tube, for redistribution and washing of the mass, which is carried out with sterilised water.

B. FULLMAN.

Purification of liquid trades waste. R. H. L. PENNELL and F. W. BRACKETT (B.P. 293,927, 29.4.27).—The liquid effluent is passed successively through a screen, a settling tank, and two or more filters, the required proportion of water for recirculation through the factory being drawn off before reaching the final filter. F. R. ENNOS.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

SEPT. 28, 1928.

I.—GENERAL; PLANT; MACHINERY.

Adhesives and adhesion. Pure chemical substances as adhesives. J. W. MCBAIN and W. B. LEE (*J. Physical Chem.*, 1928, **32**, 1178—1184; cf. B., 1927, 159).—Data are recorded for the adhesive power of many pure organic compounds when used for joining polished metal surfaces. The strength of the joint varies considerably and in relation to chemical constitution. With five specially purified, optically active higher *l*-ethylalkylcarbinols used with highly polished surfaces of steel and nickel, the joint-strength, static friction, and density are at a maximum in the middle of the series in the carbinol containing 15 carbon atoms. Stearic, palmitic, cerotic, and elaidic acids are good lubricants but poor adhesives; aurin and rosolic acid are good adhesives for metals. Pure glycollic acid after heating becomes a very strong adhesive for nickel surfaces, giving a strength of 1 ton/in.² Many pure substances give joints more stable than those of the recognised adhesives. Joints appear to fail owing to rupture of the adhesive itself and not through failure of the union between adhesive and metal. L. S. THEOBALD.

Practical pyrometry. G. B. BROOK and H. J. SIMCOX (*Inst. Metals*, Sept., 1928. Advance copy. 10 pp.).—In the aluminium industry in which currents of considerable magnitude are used adjacent to pyrometric instruments, a millivoltmeter can be protected satisfactorily against magnetic effects by shielding with soft iron plates, preferably a double shielding with an air gap. Amongst methods of protection of the thermocouple tip from attack by molten aluminium, a cast-iron sheath coated daily with a slip of chalk and graphite, using sodium silicate as a binder, was more effective than one of mild steel. A double sheath with a filling of lime was found to protect couples liable to deterioration from the attack of sulphur gases. For quick reading, bare wires were dipped into the molten metal, the metal itself giving the necessary electrical contact. The protection of base-metal thermocouples is described, and the use of lead-covered, paper-insulated cable as an extension of the couple to the recording instrument is recommended. C. A. KING.

Furnace system for chemical reactions at high temperatures. F. MEYER (*Chem.-Ztg.*, 1928, **52**, 599—600).—To obtain the advantages of a short furnace combined with heat economy, two or more small reverberatories are run in series, provision being made for direct firing of each furnace in turn and the others being preheated by the waste gases from that furnace. Alternatively, the hearth of a long reverberatory may be subdivided by means of cross-walls and the heating oil or gas supplied to each section in turn. A. R. POWELL.

Continuous extraction apparatus. H. L. MAXWELL (*Ind. Eng. Chem.*, 1928, **20**, 871).—An extraction flask containing the material to be extracted is connected with another flask by a siphon with bent-up tips and side tube for setting. Both flasks contain solvent and are heated. A vapour tube also connects them, and the excess condensation in the extraction flask operates the siphon. C. IRWIN.

Use of rotating tubes for crystallisation and evaporation. H. BARKHOLT (*Chem. Fabr.*, 1928, 431—433).—The small crystals obtained in mechanical crystallisers suffer commercially from the fact that adulteration is not so readily detectable by the eye as with large crystals. The crystal size obtained depends on the speed of rotation. Crystal forms of borax, sodium sulphide, and other salts so made are described and illustrated and costs discussed. An analogous apparatus forms an economical evaporator for the concentration of sodium sulphide solution, caustic soda, and the like, the thermal efficiency being very good. The two steps have been successfully combined in one tube with sodium and ferrous sulphates. C. IRWIN.

Recovery of vapours of volatile substances soluble in water. A. FICHOUX (*Chim. et Ind.*, 1928, **20**, 21—26).—The optimum conditions for the recovery of, e.g., acetone, when mixed with air, by absorption in water are discussed from a theoretical point of view. To remove the last traces it is more advantageous to cool the absorbing liquid than to dilute it. F. R. ENNOS.

Determination of organic solvents lost in the treatment of aqueous solutions. A. WEINDEL (*Brennstoff-Chem.*, 1928, **9**, 213—215).—The determination of the solubility of an organic solvent in aqueous residue after extraction is described. Particular mention is made of the determination of benzol in gas-works effluent liquors. A. E. MITCHELL.

Determining the densities of liquids and ore pulps. A. W. FAHRENWALD (*U.S. Bureau Mines Tech. Paper* 403, 1927, 43—51).—A device for measuring the density of sand-water mixtures comprises a thick-walled glass tube of 1 mm. bore bent at the lower end so as to form a short tube parallel to the main tube. This short stem is closed with a flat, circular, light-walled rubber bag, and both tube and bag are filled with a coloured water. On immersing the bag in a sand-water mixture so that its centre is *s* inches below the surface of the water, the liquid in the tube rises to a height *h* above the surface of the water outside. Then the density of the pulp at a depth *s* is $(h + s)/s$. To obviate errors due to eddy currents and "pockets," the mean of several readings is taken. Examples of the

use of the instrument in classification of ore pulps are given.

A. R. POWELL.

Determination of moisture by rapid drying. S. H. MEIJHUIZEN (Chem. Weekblad, 1928, 25, 494—495).—Drying for a short time at the temperature of boiling xylene (137°) gives results in many cases more accurate than those obtained by drying at 100° or 105°, decomposition being less than is usually sustained in the long drying necessary to remove all moisture at 100°. Milk powder and beet seed require only 15 min., tea only 20 min. at the higher temperature.

S. I. LEVY.

Behaviour of gases in foams. E. BIESALSKI (Z. angew. Chem., 1928, 41, 853—856).—By employment of aqueous solutions containing 0.1% of saponin and colloidal palladium, the hydrogenation of ethylene, acetylene, sodium cinnamate, and potassium oleate may be very considerably accelerated. This behaviour is contrasted with the inhibition of fire and explosion by foam formation.

S. I. LEVY.

Viscosimetry. ROWELL and FINLAYSON.—See II. **Laurent saccharimeter.** ANDERSON.—See XVII. **Dialysis of putrescible liquids.** URBAIN. **Corrosion in boilers.** GUNDERSON. **Boiler scale.** STUMPER.—See XXIII.

PATENTS.

Heat-exchange apparatus. T. W. MUNFORD, Assr. to MANTLE ENGINEERING CO. (U.S.P. 1,673,418, 12.6.28. Appl., 9.4.26).—Before the air or other gas to be heated is passed in a single circuitous stream in counter-current with the heating gases, it is conveyed, in two independent parallel streams, through the hottest part of the apparatus. The usual high mean temperature difference of the air etc. and the heating gases is thus preserved, whilst damage to the hottest parts of the apparatus is reduced and the heat radiated from the latter is more effectively utilised.

F. G. CLARKE.

Heat-exchange device. S. T. NELSON, Assr. to SULLIVAN MACHINERY CO. (U.S.P. 1,679,134, 31.7.28. Appl., 27.6.22).—A heat exchanger especially useful as an intercooler in air compression comprises a number of flat, rectangular, cooling-fluid containers, assembled parallel to each other within a casing, with detachable end supports, so that they can be removed longitudinally.

B. M. VENABLES.

Heat-treatment processes and apparatus in which a hot liquid mass [of metal] is employed. C. F. HAMMOND and W. SHACKLETON (B.P. 294,697, 2.2.27).—A bath of metal is externally heated by a fire, and kept in circulation by an air-lift effect, which may be produced by the materials to be treated, or it may be that the metal itself needs treatment, e.g., "poling." The air to the fire is preheated in spaces in the furnace walls, and the products of combustion heat a boiler in which steam is produced. The steam is superheated in a coil submerged in the metal, and part may be used to produce the air-lift effect inside a stand pipe in the centre of the bath (cf. B.P. 278,768 and 278,985; B., 1927, 928).

B. M. VENABLES.

Grinding and mixing apparatus. M. DAVID (U.S.P. 1,679,241, 31.7.28. Appl., 28.9.25. Belg.,

2.10.24).—A fixed cylindrical jacket has rotating within it a number of rollers. One roller is provided with a helical groove deepest at the feed end, becoming shallower, the latter part of the roller being smooth conical; another roller is of similar construction but reversed, and another is similar to the first except that the grooves are annular.

B. M. VENABLES.

Mixing, cooking, and drying apparatus or machines. G. PORTEUS (B.P. 294,752, 25.6.27).—In an apparatus of the type where granular material passes down through a conical chamber and is stirred by a centrally-disposed worm, the worm may be heated by a steam coil, and rotary and fixed intercalating beater bars are provided at the top of the chamber, followed by a perforated plate and by brushes. At the outlet end rotary lifters operated above adjustable segmental apertures and brushing devices similar to the upper ones may also be provided.

B. M. VENABLES.

Vertical drying apparatus. BAECHLER, KISER, & CIE. (B.P. 281,664, 29.11.27. Fr., 4.12.26).—The material falls through a series of truncated cones. After falling out of the small end of a cone it travels inwards on an intermediate plate and outwards on a main plate below, the plates being provided with scraper-blade systems which rotate or are stationary according to whether the plates themselves are stationary or rotating.

B. M. VENABLES.

Apparatus for drying or cooling material. A. G. HUNN (B.P. 294,320, 17.5.27).—The apparatus comprises fluid-heated tubes within a rotating casing which may be maintained under vacuum. The material is blown or showered by lifting blades in the casing over the tubes, which are kept clean by scrapers in the form of a cellular structure hanging loosely on the tubes and reciprocated by ropes brought through the ends of the casing.

B. M. VENABLES.

Drying of materials. J. P. DEVINE (U.S.P. 1,678,559, 24.7.28. Appl., 28.5.27).—The materials are dried under reduced pressure, in a closed heated receptacle, in the presence of a vapour which has, for a time, a relatively high humidity.

F. G. CLARKE.

Apparatus for drying industrial products. SOC. IND. DE BRIQUETERIE & CÉRAMIQUE (B.P. 275,174, 7.6.27. Fr., 30.7.26).—A drying chamber especially suitable for ceramic articles is provided with accessory apparatus such as heaters, humidifiers, fans, and dampers, all of which are controlled from one cabin.

B. M. VENABLES.

Leaching apparatus. W. L. REMICK (U.S.P. 1,678,787, 31.7.28. Appl., 17.9.25).—Solids are treated with a solution in two stages, and a filter is used from which the filtrate returns to the first stage and the filter cake to the second stage.

B. M. VENABLES.

Crystallisation apparatus. W. SEIDEL, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,679,055, 31.7.28. Appl., 16.5.27. Ger., 29.6.26).—A crystallising vessel is adapted to be rocked, and is provided with a curved bottom higher at the centre than at the sides so that when left in an extreme rocked position substantially

all the liquor will drain away from, but the bulk of crystals will remain at, the higher side.

B. M. VENABLES.

Preparation of solid bodies in a finely-divided state. BRIT. DYESTUFFS CORP., LTD., A. J. HAILWOOD, and A. SHEPHERDSON (B.P. 293,896, 11.4.27).—The powdered solid is mixed with a "comminutor" and a liquid in which both are substantially insoluble, with or without the addition of a protective colloid or dispersing agent, and the mixture is stirred or ground in a mixing machine or mill or caused to flow through pipes or over surfaces so as to produce relative motion amongst the particles. If the comminutor is of harder material than the powdered solid, the latter in a short time (15–30 min.) is reduced to particles of ultra-microscopic size. Suitable comminutors for dye powders are iron dust (which may subsequently be removed with the aid of a magnetic field), zinc powder, copper bronze, sand. When the desired degree of fineness is reached the mixture is diluted and decanted or filtered, and the dispersion may be precipitated, *e.g.*, by acids, or the solvent evaporated. The use of a dispersing agent precipitable by acids is advantageous.

C. HOLLINS.

Rotary liquid distributors. J. T. ARGENT, and ADAMS-HYDRAULICS, LTD. (B.P. 294,461, 17.5.28).—A rotary receiver from which the radial tubular arms extend is suspended at the centre of the filter bed from the middle of a diametrical tube, one half of which acts as a supply pipe. The liquid siphons over from a supply tank into the receiver, and thence to the perforated radial arms.

F. G. CLARKE.

Treatment of solid-bearing liquids. W. L. REMICK (U.S.P. 1,678,788, 31.7.28. Appl., 10.5.26).—The pulp is agitated in a first receptacle by withdrawing liquid from the top and re-introducing it at the lower part; the pulp is then settled, and a portion of the liquid withdrawn and added to the lower part of a second receptacle, in which the solids are allowed to settle on the bottom; they are then withdrawn and added to the settled solids in the first receptacle.

B. M. VENABLES.

Filtering device. W. M. MORRISON, ASSR. to AMER. SMELTING & REFINING Co. (U.S.P. 1,678,704, 31.7.28. Appl., 17.2.27).—Cakes are formed between a number of vacuum-filter leaves immersed in a tank of prefilter. The pile of leaves is withdrawn from the tank and subjected to mechanical squeezing while still maintaining the vacuum.

B. M. VENABLES.

Filters for fine filtration of liquids. O. PETERSEN (B.P. 293,668, 6.3.28).—An apparatus similar in principle to that described in B.P. 280,802 (B., 1928, 74) is constructed so that both inner and outer members have bottoms, thus becoming buckets with the walls perforated. A wide space is left between the bottoms to permit of accumulation of considerable impurity; channels lead to the space which are continuations of the slit-like openings in the walls of the buckets. In addition, a very small clearance space is left between the cylindrical surfaces, which forms the only aperture for filtrate when the filter is set as fine as possible, *i.e.*, when the slit-like apertures are entirely out of register.

B. M. VENABLES.

Pressure and vacuum filter. G. J. LIPSCOMB (U.S.P. 1,676,268, 10.7.28. Appl., 23.5.27).—The filter casing contains several layers of filter material placed on a perforate hollow carrier, inside which is a smaller imperforate tube adapted to convey liquid to one side of each layer, after which it flows away through the carrier from the opposite side of each of the layers.

H. ROYAL-DAWSON.

Apparatus for effecting intimate contact of gases and liquids. MELDRUMS, LTD., and F. MARSDEN (B.P. 294,680, 28.4.27).—An inverted, perforated, truncated cone rotates and dips into the liquid to be sprayed. Internal vanes are provided to lift the liquid, and a closed top (base) is provided for the cone to prevent the liquid passing out except through the perforations.

B. M. VENABLES.

Apparatus for cooling, cleaning, or otherwise treating gases or liquids. POWER GAS CORP., LTD., and N. E. RAMBUSH (B.P. 294,836, 19.12.27).—Nests of tubes running in one direction alternate with nests of tubes running at right angles. The sets in each direction are connected in series.

B. M. VENABLES.

Cooling of air and gases for condensing vapours therefrom. M. FRÄNKEL (B.P. 294,354, 22.6.27).—In a system of air-drying by refrigeration, cold-storage devices are arranged on each side of the refrigerator and are operated alternately in the following sequence: cooled by the dry cold air leaving refrigerator, heated and left wet by air entering the refrigerator, excess moisture blown off the surfaces by a further stream of atmospheric air, then repeat.

B. M. VENABLES.

Refrigeration [in the liquefaction of gases]. J. L. SCHLITT and W. DENNIS, ASSRS. to AIR REDUCTION CO., INC. (U.S.P. 1,678,485, 24.7.28. Appl., 20.4.25).—A gaseous mixture and the unliquefied, residual gases obtained on cooling the mixture are separately expanded, in heat-exchange relation, in a rotary expander, the resulting torque of the two expansions being applied to a single shaft.

F. G. CLARKE.

Exothermic synthesis under pressure. COMP. DE BÉTHUNE (B.P. 283,499, 10.1.28. Fr., 11.1.27).—In exothermic reactions under high pressure, *e.g.*, synthesis of methyl alcohol from carbon monoxide and hydrogen, the heat is removed actually in the reaction zone by introduction of an amount of a harmless liquid, *e.g.*, water, so small as to be negligible as a diluent, but important as a heat absorber on account of its high latent and specific heats.

B. M. VENABLES.

Carrying-out exothermic chemical reactions. L'AIR LIQUIDE SOC. ANON. POUR L'ÉTUDE ET L'EXPLOIT. DES PROC. G. CLAUDE, ASSEES. OF SOC. CHIM. DE LA GRANDE PAROISSE (AZOTE & PROD. CHIM.) (B.P. 289,823, 19.7.27. Fr., 9.8.26).—The course of the reacting gases is as follows. They enter cool through the main body of catalyst with indirect contact, then through the inner of double tubes to direct contact with a small body of catalyst, where a partial reaction without great rise in temperature takes place, back through the outer of the above-mentioned tubes, and finally through the main body of catalyst in direct contact.

B. M. VENABLES.

Apparatus for carrying out chemical reactions between gases or vapours, or gases and vapours.

H. HARTER (U.S.P. 1,678,778, 31.7.28. Appl., 27.7.26. Ger., 5.3.25).—A propeller is coated with or carries a catalytic material and serves also to aspirate and mix the gases or vapours under treatment.

B. M. VENABLES.

Apparatus for determining refraction and dispersion. H. VOELLMY (B.P. 283,859, 14.1.28. Ger., 17.1.27).—A refractometer suitable for use with ultra-violet light is described.

B. M. VENABLES.

Ascertaining the optical properties of liquids and gases. F. HERZFELD (B.P. 284,607, 30.1.28. Ger., 31.1.27).—A transparent stepped plunger is immersed in the liquid to be investigated, thus providing two different thicknesses of fluid through which the light passes; the plunger may be rotated 180° to neutralise uneven illumination, and may be made hollow so that coloured liquids may be inserted. B. M. VENABLES.

Bricks for checker work of blast and other furnace stones and regenerators. BOLCKOW, VAUGHAN, & Co., LTD., and M. A. McLEAN (B.P. 295,075, 16.5.27).

Refrigerators. W. and W. T. COLBROOK (B.P. 295,146, 30.9.27).

Evaporators for refrigerating machines. BRIT. THOMSON-HOUSTON Co., LTD., and C. STEENSTRUP (B.P. 280,538 and 293,773, [A] 7.11.27, [B] 3.5.27. U.S., [A] 13.11.26, [B] 18.5.26).

[Rotating] means for sealing vacuum treatment [drying] apparatus. O. MINTON (B.P. 283,099, 28.3.27. U.S., 26.3.26).

Tight closure or connecting means for tubes or receptacles for fluids under pressure. S. G. S. DICKER. FROM AMMONIA CASALE SOC. ANON. (B.P. 294,756, 29.6.27).

[Motor support for] liquid atomisers. SIEMENS-SCHUCKERTWERKE A.-G. (B.P. 294,460, 3.4.28. Ger., 9.12.27. Addn. to B.P. 287,105; B., 1928, 658).

Dephlegmator (U.S.P. 1,678,823). Absorbent for respirators (B.P. 293,962).—See II. Tunnel kilns (B.P. 285,323).—See VIII. Thermostat (U.S.P. 1,678,889).—See X.

II.—FUEL; GAS; TAR; MINERAL OILS.

Coalfields of Scotland. Carbonisation of "Kinneil gas" coal. (Dept. Sci. Ind. Res., Fuel Res. Survey Paper No. 11, 1928, 39 pp.).—Carbonisation tests have been carried out on a 500-ton consignment of "Kinneil gas" coal, representing the entire six-foot seam; the coal had been screened from fines (below 1½ in.) and had also been freed as far as possible by picking from associated dirt partings and slack. Analysis of an average sample gave: moisture 2.3%; volatile matter less moisture 33.5%; "fixed carbon" 57.2%; ash 7.0%. If desired, the ash could be readily reduced to 4% by efficient cleaning. Carbonisation of the coal in a Glover-West vertical retort setting using 5% of steam, and with a combustion chamber temperature of 1250° gave the following yields per ton: coke 12.6 cwt.; gas 16,770 cub. ft. of 477 B.Th.U. per cub. ft. (80 therms); tar 15.0 gals.; ammonium sulphate 23.4 lb. The coke was slightly more friable than that from strongly caking

coals and contained 12–16% of breeze; its volatile matter content was 0.4–0.8%. Working with a throughput of 3.26 tons per retort per day instead of the rated amount (2.5) increased the output of therms of gas from the setting by 23%, but decreased the yield per ton by 5.2%. Tests were also carried out with 10% of steam, and one of short duration with no steam. The yields from a horizontal retort setting (charge per retort 11.5 cwt.; carbonising period 11 hrs. 4 min.; combustion chamber temperature 1350°) were: coke 13.8 cwt.; gas 12,300 cub. ft. of 574 B.Th.U. per cub. ft. (70.6 therms); tar 9.4 gals.; ammonium sulphate 26.6 lb. A hard compact coke was produced. The coal worked well in both retort settings. It also proved very suitable for carbonisation at 625° in the Fuel Research Station vertical retorts (B., 1927, 288), giving a satisfactory smokeless fuel and yields of tar and gas above the average. The coke made by either high- or low-temperature carbonisation is quite suitable for firing a Lancashire boiler. The high-temperature coke proved satisfactory for use in a suction-gas plant, but gave trouble owing to excessive clinkering in a water-gas generator. A. B. MANNING.

Examination of a Nigerian lignite and isolation of montan wax. C. E. WOOD and S. D. NICHOLAS (J. Inst. Petroleum Tech., 1928, 14, 493–501).—The lignite contains 19.5–23.7% of water, and on analysis (calc. to a 10% moisture content) is shown to contain 58.87% of volatile matter, 25.24% of fixed carbon, 5.88% of ash, and 0.72% S. The lignite, powdered to pass 30-mesh, yields up to 19.5% of bitumen (on air-dried material). Butyl alcohol is preferred as a solvent as, owing to its comparative miscibility with water, it may be used to extract undried lignite; the extraction is practically complete after 20 hrs. The crude bitumen (crude montan wax) from Nigerian lignite extracted by means of xylene has m.p. 84–88°, acid value 41.3, saponif. value 140.5, ester value 99.2, and iodine value 48.7. Montan wax extracted by means of butyl alcohol and refined in solution by means of bauxite has m.p. 95–96° and acid value 68.0; it contains 51.5% of an acid (? montanic acid), 22.5% of esters, and 26% of unsaponifiable matter, m.p. 73–76°. The acid isolated by a modification of the method of Meyer and Brod (A., 1913, i, 1152) from Nigerian montan wax has m.p. 84–85°, *M* (monobasic) 416–420. Variation in the m.p. (70–90°) of crude montan wax examined by various authors is due to the presence of free acids other than montanic acid, of esters of acids of high mol. wt., and of hydrocarbons; an alcohol and a ketonic substance have also been detected. The ash of Nigerian lignite contains 64.69% SiO₂, 7.12% Fe₂O₃, 20.67% Al₂O₃, 2.32% CaO, 0.61% MgO, 3.48% SO₃, 1.11% alkalis and loss. W. S. NORRIS.

Influence of preheating on the theoretical combustion temperatures of gaseous fuels. F. SCHUSTER (Gas-u. Wasserfach, 1928, 71, 759–761).—The theoretical combustion temperatures of hydrogen and carbon monoxide have been calculated for a wide range (0° to 1000° or 2000°) of initial temperatures of gas and air, for the theoretical amount of air, and for 20% and 40% excess of air, respectively. The relationship between

combustion temperature and preheating temperature is not quite linear owing to the variation with temperature of the degree of dissociation of the products of combustion. The combustion temperatures of hydrogen are higher than those of carbon monoxide, except over the lower ranges of preheating temperatures when a considerable excess of air is being used.

A. B. MANNING.

Auto-ignition temperatures. II. Pure compounds. H. J. MASSON and W. F. HAMILTON (Ind. Eng. Chem., 1928, 20, 813—816; cf. B., 1928, 42).—The auto-ignition temperatures of a further range of pure organic compounds have been determined in air at ordinary pressures. Composition primarily affects auto-ignition temperature, although for widely different substances it may be almost identical, whilst closely related materials may have widely different temperatures. Auto-ignition temperatures do not decrease with increasing molecular complexity. A study of the catalytic effect of surfaces showed that a given material changes in activity during use, and that the effect of different substances is not of the same order on compounds of different composition and structure. In general, the more marked the catalytic activity of the surface the higher is the auto-ignition temperature.

H. S. GARLICK.

Equilibrium conditions in the formation of hydrocarbons and alcohols from water-gas. D. F. SMITH (Brennstoff-Chem., 1928, 9, 249—250). H. TROPSCH (*Ibid.*, 250).—In reply to Tropesch (cf. B., 1928, 147) the author states that the conclusions drawn by himself (B., 1927, 593) refer only to the temperatures for which the equilibrium constants have been calculated. Apparent discrepancies between the calculated and the experimental results are due to the varying relative reaction velocities rather than to errors in the equilibria data.

Tropesch directs attention to recent calculations of the free energies of formation of some hydrocarbons (Francis, A., 1928, 478) and to the desirability of further investigation of the free energies of formation of unsaturated hydrocarbons.

A. B. MANNING.

Influence of radiant heat on the ignition velocity of gases. K. BUNTE and A. STEDING (Gas- u. Wasserfach, 1928, 71, 673—677, 701—705, 731—734).—The ignition velocities of hydrogen, carbon monoxide, methane, water-gas, producer gas, and coal gas have been determined over a range of gas-air mixtures from measurements of the dimensions of the inner cone of the Bunsen flame produced (cf. Ubbelohde and Dommer, B., 1914, 853; 1915, 411), and the velocities determined when the gases are burnt in the open air (room temperature 20°) have been compared with those obtained when they are burnt in an enclosure maintained at 700—1000°. Only hydrogen, or gases containing a high proportion of hydrogen, show an increased rate of ignition in the heated enclosure, an increase of 15% (257 to 295 cm./sec.) being observed for the hydrogen-air mixture of maximum ignition velocity (40—45% of hydrogen) with the enclosure at 900°. With a number of gases the increase in the rate of ignition of the mixtures of maximum ignition velocity is approximately proportional to their

hydrogen content. The addition of small quantities of hydrogen to carbon monoxide, however, produces a disproportionately large increase in the velocity of ignition.

A. B. MANNING.

Influence of inert gases and water-gas on the ignition velocity of technical gases. K. BUNTE and A. STEDING (Gas- u. Wasserfach, 1928, 71, 773—778; cf. preceding abstract).—The ignition velocity of coal gas is lowered by the addition of either nitrogen or carbon dioxide, the latter producing the greater effect owing to its higher sp. heat. The effect of flue gas is approximately proportional to its carbon dioxide content. Although the addition of water-gas to coal gas lowers the calorific value, it produces a considerable increase in the maximum ignition velocity. The maximum ignition velocity of a low-temperature gas from brown coal is slightly higher than that of an ordinary coal gas. The maximum ignition velocity of towns' gas is lowered by the addition of methane, and is also displaced slightly towards the weaker gas-air mixtures. The technical importance of these results, particularly with reference to the combustion of mixed gases in burners of the Bunsen type, is briefly discussed.

A. B. MANNING.

Examination of petroleum distillation from various sources. I. Preparation of a standard motor spirit and a standard kerosene. S. T. MINCHIN and G. R. NIXON (J. Inst. Petroleum Tech., 1928, 14, 477—492).—Standard paraffinic motor spirit and kerosene have been prepared from the pressure distillate from Burnmah paraffin wax, and the physical constants determined of these and of 10° fractions thereof. The wax (solid) having $d_{20} 0.8965$ (approx.), m.p. 60.5°, mol. wt. 425, and containing C 85.25%, H 14.75%, is heated in an autoclave until the pressure reaches 100 lb./sq. in., at which constant pressure the distillation is carried out; the cracking temperature rises from 400° to 455° during the operation. The distillate is redistilled. The motor spirit (b.p. up to 150°) is given acid and alkali washes and redistilled, and the distillate is also washed and distilled in steam. The refined spirit has $d_{15.5}^{20} 0.708$, aniline point 68.1°, b.p. 66—155°. The kerosene fraction (b.p. 150—285°) is washed with sulphuric acid, treated with absorbent clay, again with acid, distilled in steam, and finally treated with acid, soda, and water. The refined kerosene has $d_{15.5}^{20} 0.764$, aniline point 82.5°, b.p. 157—292°. The motor spirit and the kerosene are redistilled through a ring-packed Hempel column, 10° fractions being taken; the physical constants indicate that the motor spirit and the kerosene are practically purely paraffinic, and that side-chain paraffins greatly predominate.

W. S. NORRIS.

Viscosimetry [of motor fuels]. H. S. ROWELL and D. FINLAYSON (J. Inst. Petroleum Tech., 1928, 14, 402—433, 444—445).—The viscosities, densities, fluidities, and "kinematic" fluidities (absolute fluidity \times density) at 10° intervals up to 50° are given for 12 motor fuels. Fluidity and more especially kinematic fluidity are much more nearly linear functions of temperature than is viscosity. In chemically related fuels fluidity is at least an approximate indication of volatility. Fluidity-sp. vol. curves are very nearly linear, and are

grouped according to chemical similarity of the fuels; aromatic hydrocarbons form one group, paraffins another, whilst commercial spirits occupy intermediate positions, "benzol" mixtures falling nearer the aromatic group than No. 1 or No. 3 spirits. Thus viscosity data and density data taken together afford evidence of the chemical composition of a fuel. The conditions governing flow of fuel through the carburettor are discussed. The viscosities of seven lubricating oils (including castor oil) have been measured up to 250° using Bingham and Michell viscosimeters. To obtain constant values 15–25 hrs. heating at 200° is necessary. Log viscosity-temperature curves of different oils are roughly parallel, almost non-intersecting, and devoid of characteristic differences. The linear fluidity-temperature relationship suggested by Bingham for "ideal" fluids does not hold for lubricating oils. For high temperatures the square root, for low temperatures the cube root, of the fluidity is in linear relation with the temperature. For extrapolation to high temperatures the square root and cube root relations may be combined using known values for lower temperatures. A new viscosity characteristic is suggested, constructed by plotting on a temperature base the viscosity multiplied by $\{(T+10)/100\}^3$, where T is temperature in °C. If an oil had fluidity (ϕ) varying according to $\phi^{1/3} = A(10 + T)$, the characteristic would become a horizontal straight line. The principles of the air-bubble viscosimeter are considered in detail.

W. S. NORRIS.

Viscosimetry of lubricating oils. II. WO. OSTWALD and A. FÖHRE (Kolloid-Z., 1928, 45, 266–279).—Prolonged mechanical treatment of lubricating oils produces changes in the viscosity, often resulting in an increase. Examination of the time effect showed that the viscosity first falls, then suddenly increases, and is followed after long treatment (120 hrs.) by a further fall. The alteration in the colour of the oil indicates that some part of the effect may be due to chemical changes such as oxidation and polymerisation. Addition of acetic or oleic acid lowers the viscosity of lubricating oils; on the other hand, small amounts of alcoholic potassium hydroxide, sodium hydroxide (but not ammonia), or aniline raise the viscosity considerably. In the case of some Voltol oils, this effect is so great that 0.28% of potassium hydroxide causes Voltol 17 to gelatinise in a few minutes. Small quantities of highly dispersed graphite and talcum, soap, and lanoline raise the viscosity of all the mineral oils examined. The Voltol oils are again exceptional, for soap lowers their viscosity, and the other agents have no effect. The gelatinising effect of potassium hydroxide on Voltol oil cannot be connected with the formation of soap from the fatty acids. The individuality of the viscosity-pressure curves of the oils in presence of addition agents is borne out by the fact that the curves often cut those of the pure oils.

E. S. HEDGES.

Theory of the formation of petroleum. Composition of low-temperature tar from Tscheremchovski boghead coal. G. STADNIKOV and E. IVANOVSKI (Brennstoff-Chem., 1928, 9, 245–248; cf. B., 1927, 736).—The fraction volatile in steam of a low-temperature tar from a Siberian boghead coal has been shown to consist

principally of oxygen and sulphur compounds, and of saturated and unsaturated aliphatic hydrocarbons; it contained only 0.9% of bases and 3.9% of phenols. The non-volatile residue, consisting of about 79% of the tar, contained about 3.5% of "asphaltenes," 22% of solid paraffins, 1.85% of bases, and 1.67% of phenols. The results support the hypothesis of the formation of boghead coal from the fats and waxes of algae, the cellulose and proteins of which have completely disappeared. They also throw light on the chemical processes undergone by such fats and waxes during geologic periods.

A. B. MANNING.

Dew points of air-gasoline mixtures from distillation curves. O. C. BRIDGEMAN (Ind. Eng. Chem., 1928, 20, 821–826).—Gasoline was vaporised by a dynamic method in the presence of known amounts of air at temperatures permitting successive approach to complete vaporisation. For each mixture and gasoline, the temperature-percentage evaporated curve extrapolated to 100% gave the dew-point temperature. Results obtained were in agreement with those of previous observers using different methods. The dew-point temperatures, at 1 atm. pressure, of gasoline vapour and of mixtures from 1:1 to 30:1 were found to be simply related to the 90% A.S.T.M. distillation points, corrected for loss, and to each other. An alignment chart given for calculating the dew-points obviates the use of the derived equations.

H. S. GARLICK.

Effect of volatility of petroleum fractions on detonation value. J. C. GENIESSE and H. F. HUF (Ind. Eng. Chem., 1928, 20, 794–796).—Detonation tests on fractions of naphthas from different sources showed a relationship between the sp. gr. and detonation value. The 50% (including loss) distillation temperature also gives a good indication. By means of a graph constructed from these data, the effect of changing the volatility on the detonation value can be predicted.

H. S. GARLICK.

Solvents in aqueous solutions. WEINDEL.—See I.
Carbon blacks and rubber. BEAVER and KELLER.—See XIV.
Petroleum oil as plant stimulant. DE ONG.—See XVI.

PATENTS.

Drying of coal and like fuels. H. FLEISSNER (U.S.P. 1,679,078, 31.7.28. Appl., 25.4.27. Austr., 14.5.26).—Coal of a colloid character is prevented from falling to pieces by first heating it under pressure in saturated steam to a higher temperature than is required to dry it, then reducing the partial pressure of the steam preparatory to quick drying.

H. ROYAL-DAWSON.

Manufacture of [smokeless] coal briquettes. G. A. CHAPMAN and E. W. WILKINSON, Assrs. to MINERALS SEPARATION NORTH AMER. CORP. (U.S.P. 1,678,379, 24.7.28. Appl., 17.3.26).—The baked briquette consists of uncoked anthracite coal passing 10-mesh and a residual oil (less than 10% on the weight of coal) as binder.

H. ROYAL-DAWSON.

Manufacture of synthetic carbonaceous coke briquettes for metallurgical use. B. E. CROCKER (U.S.P. 1,676,729, 10.7.28. Appl., 22.1.25).—The carbon residue from manufacture of oil-gas from liquid hydro-

carbons is incorporated with a gas-liberating substance, briquetted, and heated to liberate the gas.

H. ROYAL-DAWSON.

Coke ovens. C. OTTO & Co., G.M.B.H. (B.P. 287,885, 28.3.28. Ger., 28.3.27).—To avoid injurious pressure differences within the chambers of large ovens and to obtain an unobstructed discharge of the gaseous distillation products, the free space above the charge is enlarged towards the gas-discharge outlet, either by a suitable construction of the roof or by lowering the level of the surface of the charge. A. B. MANNING.

Coke oven. T. G. KUS, ASSR. to CHICAGO TRUST CO. (U.S.P. 1,676,736, 10.7.28. Appl., 16.7.23).—The oven consists of a series of elongated chambers having a wall common to each pair, each heating wall having heating chambers on opposite sides of the central plane. Ignition chambers are placed at the top and bottom of the heating chambers, with ports for conveying air to, and waste gases from, the lower members. Vertical passages between the chambers supply fuel gases to the lower ends, whilst the upper ends are supplied from the mains, which also supply the vertical passages placed at an intermediate height. Ports communicate with the upper chambers for supplying air and allowing the waste gases to escape. H. ROYAL-DAWSON.

Destructive hydrogenation of coal, tars, mineral oils, etc. I. G. FARBERIND. A.-G. (B.P. 272,539, 10.6.27. Ger., 11.6.26).—The material to be hydrogenated is supported on a porous mass of glass, quartz, earthenware, etc., up through which the hydrogen is forced in such a manner as to obtain very intimate contact between the gas and the material under treatment. A. B. MANNING.

Conversion of coaly materials into valuable liquid products. I. G. FARBERIND. A.-G. (B.P. 270,698, 30.4.27. Ger., 5.5.26).—Fine coal is mixed with a mineral oil, tar, or similar hydrocarbon, and with a finely-pulverised compound of molybdenum, and the mixture is distilled. A. B. MANNING.

Treatment of carbonaceous absorbent material for use in respirators and the like. N. C. DEAN (B.P. 293,962, 18.6.27).—Coke, vegetable carbon, etc. is treated with a solution of sodium tungstate and phosphate, dried at 150°, and immersed in fused caustic alkali, whereby its inflammability is reduced. F. R. ENNOS.

Gas producers. D. J. SMITH (B.P. 294,373, 20.7.27).—The main grate at the bottom of a vertical receptacle which acts as a hopper is frusto-conical in shape, and at its lower reduced end is a smaller frusto-conical grate pointing upwards. A double frusto-conical baffle, similar in shape to the grates, is placed immediately below the latter. The top of the inner portion of the baffle is open, to allow the gases to escape in a downward direction, the grates being slotted, but not the baffle. The air is superheated as it is sucked towards the grates down an annular space between the hopper and an outer packed chamber used for scrubbing the producer gases. All condensable gases are cracked, since they pass through a region of maximum temperature. F. G. CLARKE.

Splitting of acetylene. HYDROCARBON A.-G. F. CHEM. PROD. (B.P. 267,963, 18.3.27. Ger., 20.3.26).—The mixture of hydrogen and carbon produced in the splitting cylinders is passed through a carbon collector, and the hydrogen is then circulated repeatedly through the splitting cylinder and the carbon collector until the whole of the soot has been transferred from the former to the latter. A short pipe in which a plug of soot forms is connected to the exit of the carbon collector in order to prevent the entry of air into, or the escape of hydrogen from, the system. A. B. MANNING.

Determination of naphthalene in illuminating gas. W. H. FULWEILER, ASSR. to U.G.I. CONTRACTING CO. (U.S.P. 1,678,591, 24.7.28. Appl., 29.6.22. Cf. U.S.P. 1,443,330; B., 1923, 259 A).—The capacities of the pair of conductivity cells described in the main patent are capable of being varied from the exterior of the cells, so that the cell constants can be adjusted to equality. A. B. MANNING.

Cracking of petroleum. L. J. GARY, ASSR. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,676,895, 10.7.28. Appl., 14.3.21. Renewed 20.2.28).—The apparatus employed consists of a number of tube furnaces each of which discharges oil into an expansion chamber from which vapour passes to a dephlegmator. A pump is interposed between each expansion chamber and the associated dephlegmator. A common header joins the vapour spaces of the expansion chambers, and a second header is in communication with each dephlegmator; thus a uniform pressure is maintained in the dephlegmators, lower than the uniform superatmospheric pressure in the expansion chambers. W. S. NORRIS.

Pyrogenesis of petroleum products. F. A. HOWARD, ASSR. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,676,694, 10.7.28. Appl., 2.2.26).—Cracking stock is heated to an active cracking temperature and passed into a primary drum in which the temperature is somewhat lower, but is nevertheless above cracking temperature, and thence to a secondary drum of similar form wherein the temperature is still lower. Part of the treated oil from the secondary drum is recirculated directly into the primary drum. W. S. NORRIS.

Pressure cracking treatment of liquid hydrocarbons. ANGLO-PERSIAN OIL CO., LTD., E. S. L. BEALE, G. H. COXON, and A. E. DUNSTAN (B.P. 293,889, 15.3.27).—In the cracking of hydrocarbon oils the production of carbon is minimised by imparting surges to the liquid while maintaining nevertheless a feed flow of relatively low velocity. This is accomplished by connecting opposite ends of a double-acting, valveless, reciprocating pump to points which are respectively near the inlet and outlet ends of the heating system through which the oil is forced at a comparatively low velocity by the ordinary feed pump. Thus a rapid and violent oscillatory movement is imposed on the normal flow of the oil. In an actual experiment the feed flow was 1/30—1/20 ft./sec. The surge pump was run at 120 r.p.m., and had a 24-in. stroke. The column of oil, therefore, had imparted to it a velocity of about 12 ft./sec. alternately in opposite directions. From Persian gas oil 23—25% of motor spirit of b.p. below

200° was obtained, together with less than 0.01% of coke, and from Persian 65% residue 15% of similar spirit together with less than 0.25% of coke.

W. S. NORRIS.

Cracking [of hydrocarbon] oils. R. T. POLLOCK, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,676,924, 10.7.28. Appl., 11.4.21. Renewed 15.3.28).—Oil from a furnace is vaporised by passing it through a coil or pipe located within a vaporising chamber (external to the furnace), there being outlets from the pipe at different points along its length through which the oil passes into this chamber.

W. S. NORRIS.

Cracking of mineral oil. J. M. SCHOONMAKER, JUN., Assr. to TEXAS Co. (U.S.P. 1,678,126, 24.7.28. Appl., 15.5.22. Renewed 3.6.25).—The oil passes under pressure through a heater, cracking chamber, and separating chamber, all of which are independently heated and of progressively increasing diameter. Cracking is inhibited by applying pressure until the normal cracking range, at 100 lb./in.², is raised 6–28°.

F. G. CLARKE.

[Cracking] treatment of oils. F. A. HOWARD, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,676,826, 10.7.28. Appl., 18.5.21).—Residues are heated to cracking temperature under sufficient pressure to maintain a liquid phase, and are passed to an expansion chamber wherein volatile constituents are vaporised and removed. Fresh cracking stock is brought into heat-exchange relationship first with the conversion products removed at the lower pressure from the expansion chamber, and then with the conversion products at the higher pressure before they enter the expansion chamber. Vapour evolved from the fresh cracking stock is removed, and the residue passed to the furnace and subjected to the process as described.

W. S. NORRIS.

Manufacture of hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 293,887 and Addn. B.P. 293,906, 10.3.27).—(A) Liquid hydrocarbons obtained from the destructive hydrogenation of carbonaceous materials are converted into products rich in aromatic hydrocarbons by heating them, either alone or together with water vapour or hydrogen, in the presence of dehydrogenating catalysts. Oxides of the metals of the sixth group of the periodic system, or mixtures of these with oxides of the metals of the third and fourth groups, form suitable catalysts. Active charcoal, either alone or mixed with these metallic oxides, may also be used for the purpose. (B) The above process may be applied to hydrocarbons resulting from cracking processes, or to any hydrocarbons containing *cycloparaffins*.

A. B. MANNING.

Manufacture of gaseous olefines and liquid hydrocarbons from tars, mineral oils, and similar hydrocarbons. I. G. FARBENIND. A.-G. (B.P. 269,593, 19.4.27. Ger., 16.4.26).—The vaporised tar or mineral oil is mixed with steam and passed through a tube maintained at 700–800°, the process being carried out in the absence of metals which decompose superheated steam or which catalyse the reaction $\text{CO} + \text{H}_2\text{O} \rightarrow \text{CO}_2 + \text{H}_2$. Copper, silver, or alloys of chromium and nickel form suitable materials for the walls of the reaction vessel.

A. B. MANNING.

Production of oxidised products [from hydrocarbons]. R. B. MARTIN, Assr. to MINERALS SEPARATION N. AMER. CORP. (U.S.P. 1,678,403, 24.7.28. Appl., 30.10.20).—A continuous stream of hydrocarbons in the vapour state, mixed with oxygen, is conveyed past a source of ultra-violet light, and the oxidised products are condensed.

F. G. CLARKE.

Distillation of hydrocarbon oils. [A, c] R. D. HUNNEMAN, [B] R. E. WILSON, Assrs. to STANDARD OIL Co. (U.S.P. 1,676,609–1,676,611, 10.7.28. Appl., [A] 12.12.24, [B] 22.12.24, [C] 27.3.25).—(A) Oil is heated in a tube furnace and passed into an evaporating chamber. The unvaporised portion is passed counter-current to a stream of steam. Part at least of the steam and the vapour carried with it is forced into the stream of oil flowing through the furnace. (B) Oil is passed through a tube furnace, and thence to an enlarged vaporising chamber. Vaporised oil is removed from the latter and passed to a condenser. The unvaporised portion is subjected to contact with steam in a separate vessel, the resulting mixture of steam and vapour being then introduced at an intermediate point in the heating system. A sufficient head of liquid is maintained in the discharge line from the first vaporising chamber to prevent steam or vapour from being blown back that way. (C) Oil is heated in a tube still and discharged into an evaporating chamber. The unvaporised portion is treated with open steam, and the vapour so formed mixed with that from the vaporising chamber and carried to a vertical condenser, the reflux so formed being collected separately.

W. S. NORRIS.

Distillation [of hydrocarbon oils]. A. E. BECKER, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,676,724, 10.7.28. Appl., 6.3.23).—The oil is distilled in bulk, at a temperature not greatly above the b.p., in contact with steel wool.

W. S. NORRIS.

Sludge treatment. L. B. SMITH and G. W. JAMISON, Assrs. to ATLANTIC REFINING Co. (U.S.P. 1,676,294, 10.7.28. Appl., 9.11.25).—Sludge resulting from the treatment of petroleum with a compound of lead is treated with a substance to break the emulsion of oil and water. The aqueous layer is removed and the oil burned, whereby a solid residue is left of relatively high lead content.

W. S. NORRIS.

Reclaiming [naphthenic] distillation residues. S. P. COLEMAN and W. S. HUGHES, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,676,687, 10.7.28. Appl., 7.2.24).—Hot naphthenic distillation residues are discharged on to running water, and the oil which separates from the aqueous metal naphthenates is taken off in stages as the separation progresses.

W. S. NORRIS.

Bleaching of mineral oils and fats. I. G. FARBENIND. A.-G. (B.P. 274,828, 7.6.27. Ger., 21.7.26).—To accelerate the action of hypochlorite bleaching liquors, a salt of nickel, copper, or cobalt, an acid salt of one of the alkali metals, or a mixture of one of the former with one of the latter is added.

F. G. CLARKE.

Flotation oil. G. L. ADAMS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,678,312, 24.7.28. Appl., 28.7.27).—The oil consists of 80% of pressure-still tar,

about 19% of residuum from hydrolysis of sulphonated olefines, and about 1% of the water-soluble sodium salt of sulphonated petroleum hydrocarbons.

H. ROYAL-DAWSON.

Fuels for internal-combustion engines. C. J. SIMS (B.P. 294,129, 14.4.27).—To increase the rapidity of ignition of the fuel in compression-ignition engines, a small quantity of a primer, which has the property of exploding when rapidly heated, is added to the fuel oil. Suitable compounds for this purpose are trinitroresorcinol, methyl nitrate, iodoform, hydrazine perchlorate, etc.

A. B. MANNING.

Circulating dephlegmator [for oil refining]. D. PYZEL, Assr. to SIMPLEX REFINING Co. (U.S.P. 1,678,823, 31.7.28. Appl., 6.11.19).—Oil vapours are passed through a number of dephlegmators in series and the condensate from one dephlegmator is introduced to the top of another dephlegmator.

B. M. VENABLES.

Installations for dry-cooling of coke [in trucks]. B. E. D. KILBURN. From SULZER FRÈRES SOC. ANON. (B.P. 294,851 and Addn. B.P. 294,863, [A] 31.1.28, [B] 13.3.28).

Production of ethylene (B.P. 294,787).—See III. **Pyrolygneous acid** (U.S.P. 1,678,256).—See VII.

III.—ORGANIC INTERMEDIATES.

Miscible carbon disulphide. W. E. FLEMING and R. WAGNER (Ind. Eng. Chem., 1928, 20, 849—851).—When 55 pts. of castor oil are saponified for 2 hrs. with 10 pts. of 25% alcoholic potash at 93°, and the product is cooled and stirred with carbon disulphide until homogeneous (35 pts. of carbon disulphide to 65 pts. of the soap by vol.), the product is a mobile transparent liquid (d_{45}^{15} 1.1156) which mixes with water in all proportions forming a white emulsion, and is of use for destroying Japanese beetle without causing serious damage to plants.

F. R. ENNOS.

Introfiers, or impregnation accelerators. M. DARRIN (Ind. Eng. Chem., 1928, 20, 801—804).—Introfiers are added to impregnating materials, e.g., sulphur, in order to change the fluidity and specific wetting power of the latter towards the substance being impregnated, thus affording improved penetration. The molecular structure required by sulphur introfiers is to be found in compounds having a polycyclic nucleus, e.g., naphthalene, anthracene, diphenyl. Details of the impregnation of fibre-board with sulphur using various introfiers are given. The application to other impregnating materials than the one studied is discussed. Introfaction is closely related to solubility, and is attributed to the breaking down of associated molecules in the liquid phase.

S. S. WOOLF.

Recovery of volatile substances. FICHOUX. **Behaviour of gases in foams.** BIESALSKI.—See I. **Analysis of glycerin.** BERTH.—See XII.

PATENTS.

[Catalytic] production of ethylene. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 294,787, 27.8.27).—Acetylene is passed with an equal volume, or even with excess, of hydrogen in fine bubbles into an

inert liquid (deca- or tetra-hydronaphthalene) containing the catalyst (palladium or nickel on kieselguhr) in suspension, preferably at about 150° and under pressure if desired. Ethylene with very little ethane is produced.

C. HOLLINS.

Manufacture of formates. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 294,396, 19.9.27).—Formates of cyclic compounds are prepared by the action of carbon monoxide on cyclic alcohols in the presence of ethoxides at high pressure and at an elevated temperature. Examples given are cyclohexanol and benzyl alcohol.

C. IRWIN.

Oxidation of ethyl alcohol. HOLZVERKOHLUNGS-IND. A.-G., and O. FUCHS (B.P. 294,037, 14.11.27).—Alcohol, which may contain as much as 30% of water, is passed with air over a catalyst (e.g., silver gauze), water and acetic acid are removed by cooling, and acetaldehyde is extracted from the residual gas with an anhydrous solvent (e.g., aldol or acetic acid). The condensate is treated with sulphuric acid and ethyl acetate is recovered.

C. HOLLINS.

[Manufacture of] sulphuro-anhydride compounds of tertiary bases. E. G. BECKETT, J. E. G. HARRIS, B. WYLLAM, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 294,507, 25.2.27).—For the preparation of pyridine-sulphuric anhydride, $C_5H_5N \cdot SO_3$, and like compounds, the components (tertiary base and sulphur trioxide, or chlorosulphonic acid or esters) are brought together in the form of finely-divided mist or vapour, with or without a diluent gas, in a cooled reaction chamber, which may be rotated or agitated or may be fitted with revolving vanes. Suitable apparatus is described. The products are obtained as loose, white powders, convenient for storing and handling, and are used for sulphonations and for the preparation of sulphuric esters of leuco-compounds.

C. HOLLINS.

Manufacture of methyleugenol. W. G. ANDREW-ARTHA (U.S.P. 1,678,416, 24.7.28. Appl., 20.10.27. Austral., 29.12.26).—The mill-waste and toppings of Huon pine timber are treated with steam under pressure in a closed vessel for 2 hrs., the vapour is drawn off and condensed, and water is separated from the condensate.

B. FULLMAN.

Manufacture of calcium and magnesium salts of eugenol. V. H. KIRKHAM and L. W. RAYMOND (B.P. 294,735, 24.5.27).—Eugenol (or oils containing it) is treated with the corresponding solid oxide or hydroxide of the metal.

B. FULLMAN.

Manufacture of aliphatic and hydroaromatic sulphononic acids. I. G. FARBENIND. A.-G. (B.P. 272,967, 21.6.27. Ger., 21.6.26).—Sulphononic acids (and their salts) obtained by sulphonation of aliphatic or hydroaromatic hydrocarbons with sulphur trioxide at temperatures up to 100°, are wetting-out, emulsifying, cleansing, and solubilising agents unprecipitated by acids or by calcium or magnesium salts. In the example paraffin oil is sulphonated.

C. HOLLINS.

Manufacture of N-diaryl[di]sulphonyl derivatives of arylaminesulphonic acids. BRIT. DYE STUFFS CORP., LTD., and A. J. HAILWOOD (B.P. 293,781, 12.4.27).—Compounds of the type $SO_3H \cdot Ar \cdot N(SO_2Ar)_2$, obtained

by the action of arylsulphonyl (toluene-*p*-sulphonyl) chlorides on arylaminesulphonic acids (sulphanilic acid, metanilic acid), are useful tanning agents and assistants in the preparation of finely-divided dyes and pigments.

C. HOLLINS.

Manufacture and application of aroylating agents.

BRIT. DYESTUFFS CORP., LTD., H. M. BUNBURY, and A. SHEPHERDSON (B.P. 293,924, 26.4.27).—Benzotrichloride (1 mol.) and benzoic acid (1 or 3 mols.) are heated in nitrobenzene or other suitable solvent at about 160° in presence or absence of zinc chloride to give a solution of benzoic anhydride or benzoyl chloride which is used for benzoylation of aminoanthraquinones or other aromatic amines. The reaction is applicable to other aromatic carboxylic acids and the corresponding trichlorides.

C. HOLLINS.

Manufacture of 2:3- and 2:5-dichloro-4-acetamido-1-methylbenzene and of pure 2:3- and 2:5-dichloro-4-amino-1-methylbenzene [2:3- and 2:5-dichloro-aceto-*p*-toluidides and -toluidines]. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 294,078, 24.1.28).—2-Chloroaceto-*p*-toluidide is dissolved in glacial acetic acid and precipitated by addition of water. On chlorination, dissolution occurs and is succeeded by precipitation of 2:5-dichloroaceto-*p*-toluidide, m.p. 140—141°; the isomeric 2:3-compound, m.p. 114—115°, is obtained by dilution of the filtrate. Hydrolysis with 40% sulphuric acid converts these into the corresponding pure dichloro-*p*-toluidines, m.p. 93° (2:5) and 58—59° (2:3), respectively.

C. HOLLINS.

Production of intermediates and [wool] dyes.

L. J. HOOLEY, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 294,672, 29.1.27).—The leuco-compounds (dry or paste) of indigoid or anthraquinone derivatives not containing cyclic substituents are sulphonated, and, if desired, oxidised in the same operation, with oleum. The oxidised products are acid wool dyes. Quinizarin-sulphonic acid (orange), 1:4-diaminoanthraquinone-sulphonic acid (purple), 1:4-di(methylamino)anthraquinonesulphonic acid (blue), thioindigosulphonic acid (red), and dibenzanthronesulphonic acid (reddish-blue) are described. In many cases brighter shades are obtained than by direct sulphonation of the oxidised substance.

C. HOLLINS.

Manufacture of *p*-[4'-]amino-*o*-benzoylbenzoic acid. NEWPORT Co. (B.P. 282,001, 29.8.27. U.S., 8.12.26).—See U.S.P. 1,654,290; B., 1928, 224.

Finely-divided solids (B.P. 293,896).—See I. Oxidised products (U.S.P. 1,678,403).—See II. Glycerol (U.S.P. 1,678,150).—See XVIII.

IV.—DYESTUFFS.

Use of buffers in the determination of colour [in azo dyes] by means of titanium trichloride. O. L. EVENSON and D. T. MCCUTCHEN (Ind. Eng. Chem., 1928, 20, 860—862).—The use of hydrochloric acid in the determination of azo dyes with titanium trichloride is objectionable owing to the indistinct end-point and its limited applicability to dye mixtures. It has been found, however, that the acid can be replaced by certain salt solutions. These are: for Amaranth

sodium citrate, sodium bicarbonate, and potassium antimony tartrate; for Ponceau 3R the first two; and for Orange I the same two compounds along with sodium tartrate. The correct buffer must be determined for each dye. Results compare well with those of complete analyses.

C. IRWIN.

PATENTS.

Production of insoluble colouring materials in a state of fine dispersion. I. G. FARBENIND. A.-G. (B.P. 270,293, 25.4.27. Ger., 28.4.26).—For the dispersion of inorganic pigments "trihydroxyethylamine" is used in conjunction with glue, gelatin, casein, agar-agar or other gelatinisable substances or water-soluble gums.

C. HOLLINS.

Manufacture of dyes containing chromium. I. G. FARBENIND. A.-G. (B.P. 269,522, 4.4.27. Ger., 15.4.26).—In the conversion of chromable dyes into their chromium derivatives, less than 1 atom of chromium is used for each chromable group. The shades obtained are different from those with 1 equivalent of chromium; e.g., 4-chloro-2-aminophenol-5-sulphonic acid → phenylmethylpyrazolone, + $\frac{2}{3}$ Cr (redder); 4-nitro-2-aminophenol-6-sulphonic acid → β -naphthol + $\frac{2}{3}$ Cr (black, instead of violet-brown).

C. HOLLINS.

Manufacture of a dye containing chromium.

J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 294,743, 10.6.27).—The azo dye, metanilic acid → salicylic acid, is warmed with chromium formate solution (etc.) to give a clear greenish-yellow suitable for batik dyeing, and for yellows or greens on an indigo ground.

C. HOLLINS.

Manufacture of condensation products of the benzanthrone series and of vat dyes containing nitrogen. I. G. FARBENIND. A.-G. (B.P. 294,360, 4.5.27. Addn. to B.P. 249,891, B., 1927, 647).—Ammonia is used in place of the amine or amide of the prior process. Vat dyes result from alkaline fusion of the products. Examples are: *amino-* and *diamino-3:3'-dibenzanthronyl sulphides* from the corresponding bromo- and dibromo-compounds; the former gives by alkaline fusion a violet-blue vat dye, becoming blue by treatment on the fibre with hypochlorite.

C. HOLLINS.

New azo dyes and their application. BRIT. DYESTUFFS CORP., LTD., and R. BRIGHTMAN (B.P. 294,284, 22.4.27).—4-Nitro-4'-aminodiphenyl sulphide (I) is diazotised and coupled with an amine or phenol of the benzene or naphthalene series, including carboxylic and sulphonic derivatives. In suitable instances the product is rediazotised and coupled with another component. Examples are: (I) → 2:6-naphthylaminesulphonic acid (orange on wool); (I) → 2:6- or 2:7-naphtholsulphonic acid (yellowish-red on wool); (I) → cresidine → phenyl-J-acid (violet on wool or viscose silk); (I) → *m*-phenylenediamine (yellow-brown on acetate silk); (I) → salicylic acid (reddish-brown on acetate silk). The wool dyes are fast to milling.

C. HOLLINS.

Manufacture of new azo dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 294,291, 26.4.27).—2:3-Hydroxynaphthoic arylamides (e.g., the β -naphthylamide) are coupled on the fibre with diazotised amino-

naphthol ethers (*e.g.*, 1-amino-7-naphthyl methyl ether) to give violet shades, or in substance for violet pigments.

C. HOLLINS.

Finely divided solids (B.P. 293,896).—See I. Wool dyes (B.P. 294,672).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Swelling capacity of bleached sulphite-cellulose. W. NIPPE (Papier-Fabr., 1928, 26, 501—506).—The swelling capacity is determined hygroscopically by means of an air current the moisture capacity of which is maintained by means of saturated salt solutions. Severe chemical treatment in the digestion process reduces the swelling capacity of the cellulose; drying has a similar effect. The presence of hydrocellulose and oxycellulose tends to decrease rather than increase the swelling capacity. Comparison of this and other existing methods is made, and the use of such measurements in works control is suggested. A. G. POLLARD.

Acetylation of cellulose with pyridine and acetic anhydride. K. HESS and N. LJUBITSCH (Ber., 1928, 61, [B], 1460—1462).—Previous methods of acetylating cellulose involve the use of hydrolytic agents as catalysts. The danger thus involved is avoided when cellulose fibre, after purification in the customary manner, is immersed in sodium hydroxide solution, and subsequently washed with water until free from alkali. The water is displaced by pyridine, and the material thus obtained is shaken with pyridine and acetic anhydride during 24 hrs., after which the mixture is heated at 40—45° for a lengthy period. The triacetylcellulose thus obtained resembles closely the original material, but differs from all triacetylcelluloses hitherto described by its complete or almost complete insolubility in organic media. This difference does not necessarily denote a chemical individuality. H. WREN.

Alteration of cellulose by action of bisulphites. E. HÄGGLUND and F. W. KLINGSTEDT (Svensk Kem. Tidsskr., 1928, 40, 181—188).—Pure cellulose is not readily converted into sugar by boiling with bisulphites, a maximum of 1% being so changed, the amount of sugar produced depending on the pH value of the liquid employed. The amount of α -cellulose present, however, decreases during the boiling. A material containing 96.2% of α -cellulose contained only 31% after 9 hrs.' heating in a sealed tube at 130° with calcium bisulphite solution; further heating did not appreciably diminish this amount. The statement of Hess and others (B., 1924, 88) that the rotatory power of an ammoniacal copper solution of cellulose obtained from the acetate was identical with that of a similar solution of ordinary cellulose was confirmed experimentally. It is therefore concluded that the copper complexes formed by alkali-soluble cellulose and α -cellulose must be identical, and that any difference between these two forms of cellulose is physical rather than chemical. H. F. HARWOOD.

Testing artificial silks by means of ultra-violet light. M. KOPITSCH (Kunstseide, 1928, 10, 321—324).—It is possible to distinguish different types of artificial silk and real silk by means of differences in their fluores-

cence in ultra-violet light. Viscose silk shows a sulphur-yellow fluorescence (somewhat darker for non-purified silk) characterised by bluish gradations due to deflection of visible violet light passing through the filter of the ultra-violet lamp. Cuprammonium silk (Bemberg) shows a pink cloudy fluorescence having strong blue to bluish-violet gradations. Chardonnetsilk gives a flesh-coloured fluorescence, and is thereby easily distinguished from viscose silk. Cellulose acetate silk shows a very deep bluish-violet to violet fluorescence having very strong bluish-violet gradations. The fluorescence of artificial silks is not affected by extraction with light petroleum, but after extraction with 96% alcohol the fluorescence of cellulose acetate silk is diminished and greyish-yellow. Cuprammonium and viscose silks are not affected by scouring with a solution containing 0.5% of soap and 0.5% of ammonia; after similar treatment the fluorescence of cellulose acetate silk is paler and less blue. The fluorescence of viscose silk is not affected, that of cuprammonium silk is changed to grey and reddish-violet, and cellulose acetate silk yields a pale yellowish fluorescence after treatment with caustic soda of d 1.008. Degummed real silk shows a bright bluish fluorescence which is always more intense than that of artificial silks. The fluorescence of raw silk is largely influenced by its colour in daylight; for instance, yellow Italian grège shows a bright yellow fluorescence. A. J. HALL.

Chemical decomposition of wood. B. HOLMBERG (Papier-Fabr., 1928, 26, 506—508).—Experiments on the "mercaptolysis" of wood are described. The lignin compounds isolated from pine wood by heating with acid solutions of thioglycollic acid are described. Evidence of the presence of two types of lignin in pine wood is obtained. A. G. POLLARD.

Pulping of hardwood and mixtures of hardwoods and conifers by the sulphite process. W. H. MONSSON (Pulp and Paper Mag., 1928, 26, 705—709).—The results are given for a series of semi-commercial, experimental, sulphite cooks on aspen, sugar maple, paper birch, and black gum, alone and in admixture with white spruce and hemlock. Acid containing 6% of total sulphur dioxide (1.20% combined) was used to ensure penetration of the chips, and 2 hrs. was allowed for the cook to reach 110°, otherwise temperature curves previously found suitable for spruce were followed. The yields of pulp (dry pulp on dry wood) varied from 45% for maple to 50% for aspen with a bleach consumption varying from 8—11% for aspen to 15—35% for maple, depending on the duration of the cook. Wood infected with black rot is preferably avoided, since black rot is not removed by sulphite cooking and rather drastic bleaching is necessary, with a consequent reduction of yield and fibre strength. Hardwood sulphite pulps possess opacity, bulk, and a certain degree of softness. They develop considerable strength on beating and give a close smooth paper. Mixtures of spruce or hemlock with hardwoods are as readily pulped as the hardwoods themselves. D. J. NORMAN.

Kraft pulp and paper from *Pinus insignis*. L. R. BENJAMIN, J. L. SOMERVILLE, R. B. JEFFREYS, and W. E. COHEN (Austral. Council Sci. & Ind. Res., 1928, Bull. 35, 32 pp.).—Laboratory followed by semi-

commercial trials indicate that *Pinus insignis* can be converted into kraft pulp practically equal in quality to Swedish kraft. The best results were obtained by cooking the chips for 4 hrs. at 100 lb./in.² maximum pressure with 20% of active alkali (on the weight of dry chips), one third (approx.) of the active alkali being present as sulphide; the optimum ratio of liquor to dry wood was found to be 6:1. The use of a proportion of black liquor in the liquor charge is permissible, but has no effect on the yield of pulp. Under these conditions the yield of screened pulp averages 49% (dry pulp on dry wood). Estimated costs of production, however, show that under present conditions *P. insignis* kraft pulp could not compete economically with imported pulp.

D. J. NORMAN.

Introfiers. DARRIN.—See III.

PATENTS.

Dry cleaning and washing of wool, laundry goods, and the like. A. E. HATFIELD, E. A. ALLIOTT, and ACHILLE SERRE, LTD. (B.P. 293,886, 15.2.27).—The articles to be cleaned are washed in an organic solvent or other liquor containing a soap having a high solubility and emulsifying power and but little tendency to dissociate (cf. B.P. 289,582; B., 1928, 492), the soiled liquor being treated with a filter aid (cf. B.P. 266,850; B., 1927, 579), then filtered, and again used.

A. J. HALL.

Manufacture of wool substitutes. B. H. KANTAWALA (B.P. 293,161, 16.5.27).—Jute waste is sprinkled with an emulsion of ground-nut oil and, when uniformly impregnated therewith, is passed through a carding engine, optionally after admixture with artificial silk waste. The emulsion may be prepared by boiling ground-nut oil with 2½% of caustic soda and adding water until a 15% oil emulsion is obtained.

D. J. NORMAN.

Manufacture of cellulose acetate. H. J. MALLABAR, and NON-INFLAMMABLE FILM CO., LTD. (B.P. 293,724, 6.4.27).—Cellulose acetate, free from combined sulphuric acid, is obtained by the action of an acetylating agent on cotton in the presence of zinc chloride and 0.5–5.0% (calc. on the weight of cellulose) of hydrochloric acid. *E.g.*, cotton cellulose is treated with a mixture of glacial acetic acid, acetic anhydride, zinc chloride, and hydrogen chloride at room temperature for 5–10 hrs. until the cellulose is completely dissolved, the product being then precipitated with water; a cellulose acetate not easily soluble in chloroform but readily soluble in a mixture of chloroform and alcohol is thus obtained. Alternatively, the precipitated cellulose acetate may be made soluble in acetone by dissolving it in acetic acid, adding 5–10% of a mineral acid and 20–50% of water, and heating the mixture at 90–100° for 6–12 hrs.

A. J. HALL.

‡ **Preparing [viscose] artificial silk with special mechanical properties.** N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (B.P. 282,721, 11.11.27. Holl., 27.12.26).—Viscose filaments having 50% higher wet and dry tensile strength than those spun by the usual methods are manufactured by carrying the freshly spun filaments through at least 50 cm. in the coagulating

liquor (2.5 m. is preferred in one example given) before emerging into the air.

A. J. HALL.

Manufacture of artificial forms or threads fast to water and easily dyed. SOC. CHEM. IND. IN BASLE (B.P. 268,363, 24.3.27. Switz., 24.3.26).—Acetate silk which possesses all the desirable properties of ordinary acetate silk and can be dyed with ordinary dyes is obtained by spinning a mixture of cellulose acetate with 5–20% of nitrocellulose and denitrating the resulting filaments in a bath containing at least 8% of a soluble hydrosulphide at a temperature above 50°.

D. J. NORMAN.

Rayon [artificial silk] manufacture. G. A. RICHTER, ASST. to BROWN CO. (U.S.P. 1,678,354, 24.7.28. Appl., 13.7.26).—An aqueous solution of a cellulose derivative is heated, without appreciable coagulation, to reduce its viscosity immediately before spinning; the filaments from the spinning bath are delivered to a setting bath.

H. ROYAL-DAWSON.

Extraction of cellulose from vegetable products. F. G. P. LEAO (B.P. 293,219, 15.8.27).—Cellulose is prepared from, *e.g.*, bamboo, bagasse, cereal straws, jute, reeds, grasses, and many kinds of wood by soaking the crushed and disintegrated raw material for 4–9 days at, *e.g.*, 32° in the unfermented sap extracted from freshly cut, mature, banana plants. The cellulose remaining after the removal of the spent liquor (which latter may be used as a fertiliser) is washed first with cold dilute hydrochloric or sulphuric acid and then with water, and is finally bleached.

D. J. NORMAN.

Stencil sheet. S. HORII (U.S.P. 1,679,034, 31.7.28. Appl., 17.12.27).—The sheet consists of a base of fibrous material including esters of polysaccharides and squalene.

H. ROYAL-DAWSON.

Sizing fibres. H. L. BECHER, ASST. to AGASOTE MILLBOARD CO. (U.S.P. 1,678,720, 31.7.28. Appl., 22.4.26).—See B.P. 286,948; B., 1928, 363.

Continuous manufacture of cellulosic materials. E. MULLER (B.P. 294,676, 25.4.27).

Process for continuously drying cellulosic materials. E. MULLER (B.P. 294,675, 23.4.27).

Cellulose varnishes (B.P. 293,732).—See XIII.
Nutritive matter from fibrous vegetable materials (B.P. 293,779).—See XIX.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Mordanting and dyeing. VI, VII. E. ELÖD (Z. angew. Chem., 1928, 41, 14–16, 16–19).—VI. [With E. PIEPER and E. SILVA.] The X-ray diagram of silk weighted with tin salts consists of super-imposed diagrams of pure silk and tin dioxide, the diagram of the tin dioxide being more prominent relative to the silk diagram as the degree of weighting is increased, so that silk weighted with 30% of tin dioxide yields the diagram of tin dioxide only; similar phenomena are observed by determination of X-ray diagrams of pure silk covered superficially with powdered tin dioxide. It is concluded that in weighting silk with tin salts no chemical combination occurs between the protein of the fibre and the tin salts. Silk deaminated so that it contains only ⅓ths

of its usual content of nitrogen yields an X-ray diagram identical with that of pure silk. X-Ray diagrams of weighted silk show the particles of tin dioxide to be 33–37 Å. in length, thus being too coarsely dispersed to allow their removal from the fibre by treatment with $N-0.1N$ -hydrochloric acid. Silk mordanted with a solution of chromium chloride so that it contains 9.1% Cr_2O_3 yields an X-ray diagram identical with that of pure silk. Under comparable conditions of mordanting, silk retains 9.2% SnO_2 and but 2.3% Cr_2O_3 , the greater absorption of tin dioxide being ascribed to the greater hydrolysis of the stannic chloride during the washing process subsequent to the treatment of the silk with the metallic salt solution.

VII. [With E. PIPPER.] The absorption of a basic dye (Crystal Violet) by wool from solutions of initial p_H 1–12 steadily approaches a maximum at p_H 10; the absorption is constant over the range p_H 4.07–5.75, the isoelectric point being p_H 4.6. The rapidly decreased absorption in solutions of p_H 10.0 is due to an increase in the size of the dye particles, this increase being observed by means of an ultramicroscope. During dyeing under similar conditions, wool reduces and increases the p_H in dye solutions having initial p_H above or below 4.6, respectively, the absorption by wool of hydrogen and hydroxyl ions from acid and alkaline solutions being thus independent of the presence of a dye (basic or acid); tin-weighted silk and ordinary silk behave similarly. Japan "tram silk" weighted with tin phosphate and silicate absorbs about 60% less than unweighted silk of Rhodamine 3B from solutions of p_H 1–10; the weighting displaces the isoelectric point of the silk from p_H 4.1–5.1. Both wool and silk absorb basic dyes from solutions having p_H values greater or less than the isoelectric point of these fibres (cf. Briggs and Bull, B., 1923, 91 A). Complete absorption of the dye by silk does not occur in extremely dilute solutions of Rhodamine 3B. The absorption by silk of an acid dye (sulpho-rhodamine) differs considerably from its absorption of basic dyes, since absorption of the acid dye decreases rapidly when the p_H of the dye solution exceeds the isoelectric point. Silk deaminated so that it suffers a loss of $\frac{1}{3}$ th of its nitrogen content has an isoelectric point of p_H 4, and has a decreased and increased affinity for acid and basic dyes respectively (cf. Paddon, B., 1922, 411 A).

A. J. HALL.

Dyeing of gloving and clothing leathers. M. C. LAMB (J. Soc. Dyers and Col., 1928, 44, 225–229).—Nearly all glove leather is tanned by the alum process and then dyed by drumming (Gt. Britain) and by hand-brushing (the Continent); in America it is chrome-tanned. Before dyeing, the dried tanned leather must be "wetted-back," this being facilitated by the addition of about 1% (on the dry leather) of a diastatic enzyme (e.g., malt diastase) to the soaking liquor. In dyeing alum-tanned glove leather the use of natural dyewoods containing much tannin should be avoided, since this diminishes the extensibility required in the resulting dyed leather. Black shades are obtained by means of logwood and fustic on an iron mordant, or by drumming the leather for about 1 hr. at 40° with a solution containing 2.5% of gambier, 2.5% of chrome alum, and 2% of a direct black dye, then rinsing with water, and

re-dyeing with 1% Basic Black. Chrome-tanned leather washes more satisfactorily than alum-tanned leather; it may be dyed by means of natural, direct, basic, and acid dyes. Neolan dyes are suitable for dyeing chrome-tanned leather (alum-tanned leather may be first lightly retanned with basic chromium sulphate or basic chrome alum) in shades fast to washing. Clothing leathers should possess little extensibility, and can therefore be finished with mineral pigments (glove leathers cannot be finished in this manner owing to their greater extensibility). Clothing leathers are finished with a mixture containing a pigment and shellac as a binding and water-proofing agent. Lack of fastness to rubbing of suede leather is usually due to loose dyed fibres rather than to the use of loose dyes.

A. J. HALL.

"Zair" process. [Treatment of animal fibres with ozone.] R. B. BROWN (J. Soc. Dyers and Col., 1928, 44, 230–233).—The "Zair" process (cf. B.P. 242,027; B., 1926, 11) is described. Zaired wool has a strong affinity for acid, basic, direct, Soledon, and mordant dyes at much lower temperatures (40°) than are used for dyeing ordinary wool (100°). Under similar conditions of dyeing, ordinary and Zaired wool absorbed 43.9 and 71.9% at 40°, 62.5 and 94.2% at 60°, and 93.0 and 94.9% at 100°, of the total amount of Acid Magenta O in the dyebath. Zaired wool dyed at a low temperature is less penetrated by dyes than ordinary wool dyed at boiling temperature; Zaired wool yarn dyed at 40° so as to absorb 76.6% of Acid Green had a depth of shade equal to that of ordinary wool dyes at the boil so as to absorb 94.5% of the dye, but the interior fibres of the Zaired yarn were much paler than the outer fibres. After treatment by the Zair process, wool has an increased tensile strength, and, as in chlorination, the epithelial scales are partially removed. Zaired wool is superior to chlorinated wool, since it may be dyed evenly by a much wider range of dyes. The Zair process is useful for increasing the affinity of wool for dyes before printing. Cellulose fibres and real silk suffer a considerable loss of strength when treated by the Zair process; cellulose acetate silk is not appreciably affected. The dyeing of furs and sheepskins is much assisted by a preliminary treatment by the Zair process.

A. J. HALL.

Chemical effects of the natural sulphur in wool on fading of azo dyes. A. T. KING (J. Soc. Dyers and Col., 1928, 44, 233–236).—The suggestion of Meunier and Rey (B., 1926, 974) that the natural sulphur present in wool is oxidised to sulphurous acid during exposure to light has been confirmed by exposing scoured wool fabric to light from a fadeometer, then padding it with slightly alkaline solutions of Orange II and Crystal Scarlet, and plunging it into a boiling slightly acid liquor, the formation of the yellower azo-sulphite derivatives of these dyes being clearly observed on the exposed parts of the fabric. The azo-sulphite derivatives of Crystal Scarlet, Orange II, Fast Red A, and Acid Orange GG faded 150–200% more rapidly than the original dyes, but during the fading there was no evidence of conversion of the azo-sulphite into the original dye, this latter fact agreeing with the remarkable stability of these azo-sulphites towards iodine and other non-alkaline oxidising

agents. Oil colours such as Oil Orange E, Oil Vermilion, Oil Red BN, and Oil Brown D dyed on wool in the form of their azo-sulphites also faded with increased rapidity. The greater rate of fading of the azo-sulphite derivative accounts for the failure to detect formation of azo-sulphite formation when wool padded with a suitable β -naphthol azo dye is faded by exposure to light. Dyes capable of forming azo-sulphites faded more rapidly on alkaline than on acid cloth. Dyes susceptible to reduction by sulphur dioxide, *e.g.*, Fast Acid Blue RH, faded slightly more rapidly on acid than on alkaline wool. With few exceptions, dyes of poor fastness to stoving (sulphur dioxide) are also of poor fastness to light on wool.

A. J. HALL.

Action of light on dyed colours. F. SCHOLEFIELD, E. HIBBERT, and C. K. PATEL (J. Soc. Dyers and Col., 1928, 44, 236—237).—Pure crystalline dibromoisatin and 2-methylisatin have been separated from the aqueous extract of cotton dyed with Ciba Blue 2B and methyl-indigo, respectively, and faded by exposure to sunlight. From this and previous results (*cf.* Hibbert, B., 1927, 840; Haller and others, B., 1928, 479) it is concluded that oxidation is the cause of fading of cotton materials dyed with indigoid dyes.

A. J. HALL.

Determination of Katanol O and investigation of its absorption by viscose silk. P. E. KING, G. M. WADADEKAR, and E. N. JOHNSON (J. Soc. Dyers and Col., 1928, 44, 237—241).—Katanol O (*cf.* B.P. 173,313; B., 1922, 139 A) may be determined in aqueous solutions by a modification of the potassium permanganate method used for tannic acid. Dilute sulphuric acid (5 c.c. of the concentrated acid in 100 c.c. of water) is added to 10 c.c. of a solution of Katanol (1 g. of Katanol, 0.2 g. of anhydrous sodium carbonate, 500 c.c. of water) and 200 c.c. of water, whereby the Katanol is precipitated, and the product is then titrated with 0.1*N*-potassium permanganate until a pink coloration persists for 2 min. after the addition of 2 drops; the potassium permanganate solution is added only 0.5 c.c. at a time, and the suspension is heated at 70° towards the end of the titration. The presence of Glauber's salt does not affect the titration. The absorption of Katanol O by viscose silk increases with duration of mordanting process, concentration and temperature of the mordanting liquor, and the use of Glauber's salt as an assistant. Maximum absorption occurs within 2 hrs. of entering the silk, and when entered at 100° or 85° but little further absorption occurs after the first $\frac{1}{2}$ hr. The absorptions of Katanol O by viscose silk immersed for 2 hrs. at 60° in solutions of equal concentration but having vol. of liquor/wt. of viscose silk ratios of 12:1 and 50:1, were 1.76 and 0.46%, respectively; the absorptions in the presence of 0, 25, and 50% of Glauber's salt were 0.65, 1.48, and 1.83% respectively. Soda ash has little effect on the absorption, but the maximum absorption occurs when Katanol O:soda ash = 5:1. Viscose silk mordanted with Katanol O bleeds during washing; this is largely prevented by souring with dilute acetic acid.

A. J. HALL.

Rubber colours. NAUNTON.—See XIV. Coal-tar colours in foods. JAMIESON and KEYWORTH.—See XIX.

PATENTS.

Bleaching composition. E. M. JONES (U.S.P. 1,677,283, 17.7.28. Appl., 17.8.25).—The composition contains sodium silicate, oxalic acid, borax, and sodium perborate. This composition may be used in an iron kier without the formation of rust, and is capable, when used in boiling liquors, of simultaneously bleaching the white ground of printed fabrics and setting the colours.

D. J. NORMAN.

Dyeing and printing [with water-soluble vat dyes]. STANDFAST DYERS & PRINTERS, LTD., J. I. M. JONES, and W. KILBY (B.P. 293,890, 15.3.27).—Cotton, silk, and wool materials dyed or padded with water-soluble esters of vat dyes (*cf.* B.P. 247,787 and 251,491; B., 1926, 403, 625) are developed to their full shade by treatment with a boiling acid solution containing a cupric salt; over-oxidation of the dye which may occur when development is effected with nitrous acid or a dichromate is thus avoided. *E.g.*, wool dyed with the water-soluble ester of dimethoxydibenzanthrone is rinsed and then immersed in a boiling bath containing 5 pts. of copper sulphate and 10 pts. of sulphuric acid per 1000 pts. of water, development being complete within 10 min.

A. J. HALL.

Production of dyeings on the fibre. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 293,813, 6.4.27).—Cotton is impregnated with a phosgenated *p*-aminoazo or *p*-aminodisazo dye containing one or more carboxyl groups (excluding the salicylic group), and is then immersed in a solution of a chromium salt with or without an intermediate drying. The process is especially useful in batch dyeing. Examples are the phosgenated derivatives of: 2-chloro-3-amino-5-sulphobenzoic acid \rightarrow cresidine (golden-yellow); 3-amino-5-sulphobenzoic acid \rightarrow *o*-anisidine (greenish-yellow); aminoazobenzene-disulphonic acid \rightarrow 3-amino-*p*-tolylglycine (reddish-orange).

C. HOLLINS.

Dyeing of fibres. A. WINOGRADOFF, ASSR. to INECTO, INC. (U.S.P. 1,677,508, 17.7.28. Appl., 11.7.25).—When the colour is developed by the oxidation of a leuco-compound the fibres are treated with an inorganic iodide.

D. J. NORMAN.

Treatment of fibrous materials. I. G. FARBENIND. A.-G. (B.P. 267,924, 14.3.27. Ger., 18.3.26).—Mixtures of sulphonated aromatic, hydroaromatic, and saturated and unsaturated cyclic and aliphatic compounds such as may be obtained from mineral oils, oil distillates, and brown-coal tar oils, may be used as assistants in the wet processing (bleaching, dyeing, carbonising, finishing, etc.) of wool, cotton, silk, and other textile materials, or in mixture with other assistants such as soaps, Turkey-red oils, organic solvents, etc.

A. J. HALL.

Production of azo dyes on weighted silk. I. G. FARBENIND. A.-G. (B.P. 271,089, 11.5.27. Ger., 11.5.26. Addn. to B.P. 253,865; B., 1927, 553).—In the process of the prior patent when applied to weighted silk the effect of the alkaline padding bath on the weighting agents is avoided by adding to this bath a soluble silicate and/or a soluble phosphate.

C. HOLLINS.

Increasing the fastness to light of basic dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P.

294,286, 22.4.27).—Materials dyed with basic dyes (*e.g.*, tannin-mordanted cotton dyed with Rhodamine B extra) are treated with a solution of reduced phosphotungstomolybdenum compound of B.P. 292,253 (B., 1928, 603). C. HOLLINS.

Preventing the weakening of vegetable fibres in the production of Aniline Black. K. SCHMIDT (B.P. 294,554, 24.1.27).—Salts of weak or easily oxidisable acids are added to the padding liquors used for dyeing Aniline Black on cotton materials by the copper or prussiate methods in order to increase the stability of the padding liquor and the fastness of the resulting black shade, and also to diminish the tendering of the cotton fibres; the amount of protective salt used is not less than 40% (by wt.) of the aniline hydrochloride present in the padding liquor. Suitable salts are those of sulphurous, phosphorous, and thiocyanic acids.

A. J. HALL.

Process and apparatus for parti-colour treatment of yarns. J. BRANDWOOD (B.P. 294,504, 22.1. and 29.4.27).

Azo dyes (B.P. 294,284).—See IV. **Artificial threads** (B.P. 268,363).—See V. **Dye soaps** (B.P. 270,637).—See XII. **Coloured reproductions** (B.P. 270,279).—See XXI.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Assay of phosphoric acid. F. A. MAURINA (J. Amer. Pharm. Assoc., 1928, 17, 668—671).—The U.S. Pharmacopœia method of determining phosphoric acid, viz., by adding excess of silver nitrate to a solution neutralised to phenolphthalein, maintaining neutrality by addition of zinc oxide, and titrating the filtered solution with potassium thiocyanate, has the disadvantage that zinc oxide does not react completely and forms a bulky deposit in which silver may be included. The zinc oxide is therefore replaced by an aqueous suspension of the hydroxide, prepared from zinc carbonate and potassium hydroxide, by means of which accurate results are obtained. E. W. WIGNALL.

Determination of sulphur combined as sulphides in potassa sulphurata (liver of sulphur). C. H. ROGERS (J. Amer. Pharm. Assoc., 1928, 17, 658—661).—Liver of sulphur is treated in aqueous solution with excess of standard copper sulphate solution, and the precipitate is removed by filtration; the copper in the filtrate is precipitated as sulphide, which is collected on a filter and converted into copper oxide by ignition. The copper employed in the first precipitation is found by difference; it is considered to have combined with sulphide ions, and the sulphur present as sulphides is thus determined. E. W. WIGNALL.

Physical characteristics of chromium sulphate solutions. W. J. CHATER and J. S. MUDD (J. Soc. Leather Trades' Chem., 1928, 12, 272—279).— p_H value—basicity and conductivity—basicity curves derived for solutions of chromium sulphate and chrome alum, respectively, indicate a change in the constitution of the liquors at about basicity 124 and p_H 2.3. The change in p_H due to the presence of neutral salt appears to be

dependent on the basicity. The difference in acidity caused by the neutral salt is a minimum at basicity 124. The penetration of gelatin jelly by chromium sulphate solutions has been shown to depend on the acidity; the greater the acidity the greater is the penetration. D. WOODROFFE.

Analysis of pitchblende. E. KÖRNER and F. HECHT (Monatsh., 1928, 49, 438—443, 444—459).—The analytical scheme of Davis (A., 1926, 380) appears to yield trustworthy results, and with certain minor modifications has been used to obtain complete analyses of Morogoro and Katanga blends. Assuming that molybdenum is present as lead molybdate (wulfenite), the lead/uranium ratios are of the same order of magnitude as those observed by Davis and others for Katanga blende. J. S. CARTER.

Thorium content of Katanga pitchblende. F. HECHT and E. KÖRNER (Monatsh., 1928, 49, 460—475; cf. preceding abstract).—Improvements in the method for the determination of thorium according to the Davis scheme are suggested and possible sources of error are indicated. Such data as exist indicate that thorium is not, as has been suggested, a decomposition product of a uranium isotope. J. S. CARTER.

Detection of carbon dioxide. G. ELTESTE (Z. angew. Chem., 1928, 41, 858).—Filter paper moistened with a solution of 0.1*N*-barium hydroxide to which 10% by vol. of a 0.1% solution of phenolphthalein has been added is decolorised immediately in an atmosphere of carbon dioxide, due to the formation of barium bicarbonate. The colour reappears in air, and with freshly prepared damp papers the change may be repeated several times. The papers were used to detect leaks in containers of compressed gas. S. I. LEVY.

Stress-strain curves for plastic sulphur and raw rubber at various temperatures. J. D. STRONG (J. Physical Chem., 1928, 32, 1225—1230).—The stress-strain curves for thin filaments of plastic sulphur have been determined by means of a modified Jolly balance at -10.3° , -3.6° , and $+11.2^\circ$; and those for raw rubber on a modified Schopper machine at -65° , -60° , -55° , and -45° . The automatic recording of the data, by increasing the speed of testing, made the measurements less dependent on plastic flow. A certain similarity is claimed for the two series of curves. The data for rubber do not agree quantitatively with those of Kröger and Le Blanc (B., 1925, 932).

L. S. THEOBALD.

Crystallisation etc. BARKHOLT.—See I. **Reactions between ferrous oxide and carbon.** FALCKE.—See X.

PATENTS.

Manufacture of sulphuric acid. H. PETERSEN (B.P. 276,659, 15.8.27. Ger., 28.8.26).—The first production tower in a series for manufacturing sulphuric acid without lead chambers is of large dimensions, and is irrigated intensively with nitrosylsulphuric acid so that an amount of nitrogen oxides is liberated at least sufficient to oxidise the sulphur dioxide in all the subsequent towers. W. G. CAREY.

Manufacture of sulphuric anhydride and sulphuric acid. VEREIN F. CHEM. U. MET. PROD. (B.P. 289,879, 27.4.28. Ger., 5.5.27).—The contact process for the preparation of sulphuric anhydride is separated into stages with systematic arrangements for heat transfer. In one method the gases leaving the first catalyst chamber pass around the second chamber before removal of sulphuric anhydride, then through a secondary heat-exchanger to the absorption apparatus. The gases from the second catalyst chamber are made to transfer their heat to the supply for the first chamber. A conversion of 80–90% is obtained in each stage without any considerable external heat, and the process can be operated without risk on rich gas containing 8–9% SO_2 . C. IRWIN.

Simultaneous production of phosphorus or phosphoric acid and binding agents with latent hydraulic properties. I. G. FARBENIND. A.-G. (B.P. 285,055, 27.1.28. Ger., 10.2.27).—In the production of phosphorus or phosphoric acid in an electric or shaft furnace clay is added to the mixture of phosphate and coke so that a slag is obtained containing 12–35% Al_2O_3 , 18–38% SiO_2 , and 40–55% CaO . The expulsion of phosphorus is complete and there is no tendency to the formation of carbides as occurs if bauxite is used. The slag on granulation is suitable for the production of blast-furnace slag cement. C. IRWIN.

Manufacture of white lead acetate solutions and crystals from pyroligneous acid. M. KLAR (U.S.P. 1,678,256, 24.7.28. Appl., 22.12.25. Ger., 28.10.24).—Pyroligneous acid containing organic colouring impurities is neutralised with lead or its compounds, and the solid acetate formed is then heated at 200–220°, lixiviated, and the solution filtered, evaporated, and allowed to crystallise. H. ROYAL-DAWSON.

Preparation of cooling brine. N. DAHL (B.P. 281,632, 14.11.27. Fr., 1.12.26).—Salt solution flows through an ice column surrounded by a cylindrical receptacle or series of receptacles arranged co-axially within one another and fitted with horizontal and vertical baffles disposed so that the mass of liquid is divided and is caused to follow a zig-zag path. W. G. CAREY.

Preparation of sodium phosphate. H. HOWARD, ASSR. to GRASSELLI CHEM. CO. (U.S.P. 1,676,556, 10.7.28. Appl., 4.12.25).—Phosphoric acid solution containing a sulphate and a silicofluoride is treated with a sodium compound, then with a barium compound capable of precipitating the total sulphate from solution.

H. ROYAL-DAWSON.

Manufacture of phosphate fertilisers. L. ADELANTADO (B.P. 276,297, 17.1.27. Spain, 19.8.26).—Natural phosphates are treated with soluble sulphates in the proportion of 2 mols. of tricalcium phosphate to 3 mols. of sulphate in the presence of 3 mols. of mineral acid additional to that required to react with any impurities. The product is extracted with water and crystallised. The sulphates indicated in examples are ammonium and potassium sulphates. The product is a mixture of mono- and di-alkali phosphates, practically all the phosphoric acid being water-soluble. The aqueous extract obtained is slightly acid. If not neutralised and crystallised they may be employed for impregnating inert matter, e.g., peat. C. IRWIN.

Production of commercial borax from $\text{Na}_2\text{O}, 2\text{B}_2\text{O}_3, 4\text{H}_2\text{O}$. T. M. CRAMER, ASSR. to PACIFIC COAST BORAX CO. (U.S.P. 1,678,381, 24.7.28. Appl., 12.1.27).—The naturally-occurring mineral is rendered more soluble by heat, then dissolved in an aqueous medium, and commercial borax is crystallised out.

H. ROYAL-DAWSON.

Hypochlorite composition. J. D. MACMAHON, ASSR. to MATHIESON ALKALI WORKS (U.S.P. 1,678,987, 31.7.28. Appl., 20.5.27).—The dry mixture consists of anhydrous sodium sulphate and calcium hypochlorite.

H. ROYAL-DAWSON.

Production of alkali compounds from silicates containing them. T. A. EDISON, ASSR. to T. A. EDISON, INC. (U.S.P. 1,678,246, 24.7.28. Appl., 28.6.22).—The pulverised silicate containing an alkali metal is mixed with an alkaline-earth hydroxide and water, and the mixture heated sufficiently to free the alkali and then lixiviated.

H. ROYAL-DAWSON.

Production of base-interchanging substances. A. ROSENHEIM (B.P. 266,313, 27.1.27. Ger., 16.2.26).—Minerals containing iron oxide, alumina, etc., but deficient in constituents having base-exchange properties, are treated at increased pressure and, if necessary, raised temperature with solutions of alkali, alumina, or silicic acid, the last-named preferably as water-soluble silicates rich in silicic acid. Hydrolysis with water vapour is then effected (cf. B.P. 265,578; B., 1928, 110).

W. G. CAREY.

Production of aluminium oxide from aluminium sulphide or mixtures containing same. METALLBANK U. METALLURGISCHE GES. A.-G., and C. B. VON GIRSEWALD (B.P. 294,079, 27.1.28).—Aluminium sulphide is converted into the oxide by heating with calcium oxide and/or a calcium compound convertible into the oxide by heating; or the calcium oxide etc. may be added to molten aluminium sulphide. The resultant aluminium oxide is freed from the calcium sulphide formed by treatment with water and acids.

B. FULLMAN.

Production of metal cyanamides or mixtures containing the same. N. CARO and A. R. FRANK (B.P. 279,420, 30.9.27. Ger., 23.10.26).—A mixture of ammonia and carbon monoxide is allowed to act on calcium carbonate at its dissociation temperature. The carbon monoxide decomposes the water vapour and prevents reversal of the reaction. Metals which catalyse the decomposition of ammonia must not be used in the apparatus, which is best made of silica.

C. IRWIN.

Fluorescent preparations. L. A. LEVY and D. W. WEST (B.P. 295,078, 19.5.27).—Fluorescent preparations of desired colours are prepared by mixing fluorescent substances with non-fluorescent or feebly fluorescing substances of suitable shades, such as dyes, pigments, oil paints, etc.; e.g., a mixture of zinc and cadmium sulphides with vermilion oil paint gives a scarlet fluorescence.

L. A. COLES.

Exothermic synthesis (B.P. 283,499).—See I. **Bleaching composition** (U.S.P. 1,677,283).—See VI. **Fertilisers** (B.P. 283,558).—See XVI. **Organic tin compounds** (B.P. 294,287).—See XX.

VIII.—GLASS; CERAMICS.

Brown silica bricks. V. E. GROOM-GRJIMAILLO (Feuerfest, 1928, 4, 105—106).—In the manufacture of silica bricks, the inversion of quartz to tridymite is not readily attained if pure, coarsely-ground quartzites are used. The formation of tridymite is facilitated if iron-bearing material, such as furnace slag, is added to the brick mixture, together with a reducing agent (coal, coke, etc.). The slags provide the necessary lime-iron flux "ready-made," and the addition of powdered charcoal, coke, etc. ensures the formation of ferrous oxide, and not ferric oxide, the former having better solvent properties. The addition of phosphoric acid promises special advantages, but this awaits confirmation from works' practice. Bricks were made in the usual way, but with the addition to the quartzite of 2.5% of basic slag from a Martin furnace (grain-size below 1 mm.), powdered wood charcoal, and 2% of milk of lime. The bricks obtained were of a chocolate colour; under the microscope the ground mass appeared as a network of interlocking crystals, only the coarser quartz grains being shattered and unconverted. The bricks showed no expansion or cracking in service in a Martin furnace, and they proved much more durable than ordinary silica bricks, and were mechanically much stronger. The chemical composition was: 93.68% SiO_2 , 0.19% Al_2O_3 , 2.31% Fe_2O_3 , 3.21% CaO , 0.25% MgO , 0.38% MnO (P_2O_5 not determined). Similar experience was obtained with bricks made with the addition of 2—4% of reheating furnace slag, 1.5% of clay, 1.5% of coke dust, and 1—1.5% of milk of lime.

F. SALT.

[Quartz] inversion phenomena in silica bricks in the crowns of Martin furnaces. V. E. GROOM-GRJIMAILLO (Feuerfest, 1928, 4, 125).—Silica bricks taken from the crown of a Martin furnace consist of four zones, and analysis of these zones shows a considerable accumulation of lime in the third (brown) zone from the hot face. Before use, a silica brick contains colloidal tridymite in which quartz grains are embedded; these grains are, so to speak, "moistened" with the glassy lime-iron-alumina matrix. The colloidal tridymite constitutes the solid cement, binding the brick together. The quartz is converted by the glass into colloidal tridymite, which subsequently changes into the crystalline form. The more fusible the glassy matrix, the more readily does quartz inversion take place. Ferrous oxide in the furnace gases is absorbed by the bricks, and this increases the fusibility of the fluxes in the bricks. If iron is absorbed in sufficient quantity to form the eutectic compound $3\text{FeO} \cdot \text{SiO}_2 \cdot \text{CaO} \cdot \text{SiO}_2$, the glass has a softening point of 1030° , and its capacity to penetrate the brick by capillary action in an upward direction reaches a maximum. Ferrous silicate, on the other hand, has a softening point of 1190° , so that a glass of this composition remains in the lower portion of the brick. This explains the accumulation of lime in the upper portion. It is suggested that ferrous oxide and lime should be added in the form of Martin furnace or Thomas slag to the silica brick mixture, together with a certain quantity of coke or coal dust, in order to ensure the formation of ferrous silicate.

F. SALT.

Destruction of blast-furnace building materials,

particularly firebricks. E. DIEFSCHLAG and K. FEIST (Feuerfest, 1928, 4, 49—51, 106—108).—Methods for testing the resistance of refractories to slags are critically reviewed. Tests were made on a number of firebricks by filling small hollows in them with weighed quantities of sodium and potassium hydroxides, and firing to various temperatures between 200° and 1100° , the change in the alumina:silica ratio being taken as a measure of the reaction. A considerable quantity of brick material was dissolved by sodium hydroxide at 400° , though no corrosion of the refractory was evident. Silica, particularly in the amorphous form, was attacked much more readily than alumina at this temperature, but at 600 — 800° this difference was largely eliminated. Potassium hydroxide behaved similarly, but the quantity of refractory dissolved was much smaller. The melts formed at the low temperature were largely soluble in water, indicating the formation of simple silicates and aluminates, whereas those formed above 1000° took the form of insoluble complexes. Between 1000° and 1015° the quantity of brick material dissolved increased considerably, but the lower reactivity of potassium hydroxide was still more marked at this temperature. The degree of penetration into the body of the brick depends almost entirely on the viscosity of the melt produced. It therefore does not constitute a measure of the chemical reaction which has taken place. The alumina content of the refractory has no important effect on the resistance to slag attack.

F. SALT.

PATENTS.

Manufacture of glass in a shaft furnace. K. KÜNZEL (U.S.P. 1,676,267, 10.7.28. Appl., 18.3.27. Ger., 10.2.25).—A flame is carried upwards through the shaft and the charge of crude glass material contained in the furnace.

II. ROYAL-DAWSON.

Preventing the weathering (especially clouding, tarnishing, and lustering) of the surface of glass. K. KAMITA, and ASAHI GARASU KABUSHIKI KAISHA (B.P. 294,391, 25.8.27).—The surface is exposed to the action of an acid gas, or a gaseous mixture containing such, at 300 — 600° (e.g., at or near the annealing point), after which the white deposit so formed is wiped off. The gas may be introduced into thelehr during the annealing process.

A. COUSEN.

Manufacture of tinted [violet] glass. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST. GOBAIN, CHAUNY, & CIREY (B.P. 293,310, 30.6.28. Fr., 2.7.27).—Violet glass which in combination with green silicate glass forms neutral grey doublets is produced by the addition of titanium oxide to a vitrifiable mixture having a base of phosphoric acid or a mixture of it with boric acid. A suitable composition contains 72.5% P_2O_5 , 22.4% CaO , 3.1% MgO , and 2.0% TiO_2 .

L. A. COLES.

Tunnel kilns. WOODALL-DUCKHAM (1920), LTD., and A. McD. DUCKHAM (B.P. 285,323, 12.8.27. Addn. to B.P. 280,044; B., 1928, 92).—More gradual cooling of the ware is effected by connecting the exhaust ducts with longitudinal flues in the walls of the cooling zone, these flues being in communication with the interior of the kiln at a point between the ducts and the open

heating zone. In the preheating zone the products of combustion escape through groups of ports, each group communicating with a separate duct in the lower portion of the kiln wall; the duct in turn communicates with waste-gas flues in the kiln walls. F. SALT.

Manufacture of ceramic ware. L. MELLERSH-JACKSON. From AMER. ENCAUSTIC TILING Co., LTD. (B.P. 293,069, 28.2.27).—A ceramic body contains about 27% of magnesia, 6% of alumina, 61% of silica, to which a small proportion of calcium, in any suitable form, is added. For the semi-dry press method of manufacture the powdered mixture contains about 10% of water. In the manufacture of tiles, a pressure in the moulds of about 1200 lb./in.², a maximum firing temperature between cones 6A and 7, a biscuit-firing period of about 48 hrs., and a glaze-firing period of about 12–14 hrs. are required. The biscuit ware may be saturated with an organic substance to improve the dielectric properties or to render it non-absorbent to water. F. SALT.

Production of ceramic and like articles. M. HAUSER (B.P. 289,031, 26.4.27. Switz., 28.4.26. Cf. B.P. 270,300; B., 1928, 606).—A mixture of ceramic raw materials, *e.g.*, clay, felspar, quartz, etc., with powdered or granular silicon or ferrosilicon, with or without the addition of boric acid or borates and/or adhesives, *e.g.*, glucose, tragacanth, etc., is worked up into a plastic mass, shaped, and fired. L. A. COLES.

Reducing the coefficient of expansion of ceramic materials. F. SINGER (B.P. 282,403, 14.12.27. Ger., 17.12.26).—The coefficient of expansion of steatite bodies and similar ceramic materials containing magnesium or alkaline-earth compounds is reduced by the addition of an aluminium compound (hydroxide) to the body mixture and submitting this mixture to suitable heat-treatment until at least three phases are formed: a glass having n 1.530–1.555; crystals of the sillimanite or mullite type; and alkaline-earth silicates, particularly magnesium silicates, of the type RO_2SiO_2 and $2\text{RO}_2\text{SiO}_2$. The heat-treatment must be interrupted when there is risk of the formation of spinels of the type $\text{RO}_2\text{Al}_2\text{O}_3$. F. SALT.

Manufacture of silicon carbide refractory articles. CARBORUNDUM Co., LTD., Assees. of M. L. HARTMANN (B.P. 284,732, 4.2.28. U.S., 4.2.27).—Oxidation of silicon carbide at high temperatures is greatly reduced by the removal of certain catalysts, *e.g.*, the oxides or salts of easily reducible metals, particularly iron. Silicon carbide is therefore freed of such impurities, and refractory articles are made by mixing about 90% of this purified material with about 10% of a suitable bond, *e.g.*, a mixture of kaolin and felspar. F. SALT.

Manufacture of objects from zirconium [oxide]. DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT VORM. ROESSLER (B.P. 282,795, 23.12.27. Ger., 27.12.26).—Zirconia is intimately mixed with small amounts of a hydrolysable salt (*e.g.*, the chloride) of zirconium, aluminium, or magnesium, and water is then added to give a plastic mass, which is shaped and fired in the usual way. Other refractory materials, such as thoria, may partly replace the zirconia. A. COUSEN.

Manufacture of abrasives. O. Y. IMRAY. From T. HIRAO, S. SUZUKI, and T. SUZUKI (B.P. 294,124, 15.1.27).—Finely-powdered volcanic ash and obsidian (the latter heated to 900° before crushing) are mixed with small-grained quartz, sand, or other silicious material, the mixture is then pressed in a mould, and heated to fuse the obsidian and ash. A. COUSEN.

Temperature control in furnaces or lehrs. BRIT. HARTFORD-FAIRMONT SYND., LTD. From HARTFORD-EMPIRE Co. (B.P. 294,775, 4.8.27).

Drying apparatus (B.P. 275,174).—See I.

IX.—BUILDING MATERIALS.

Light porous concrete. J. MEYER (Chem. Fabr., 1928, 433–435, 449–450).—To produce a porous concrete, 0.1–0.5% of an alloy of calcium is added to the cement mixture before addition of water. Moulds are only filled, to the extent of 70–80% and the evolution of gas can be regulated to require 30–45 min. or 3–4 hrs. The concrete can, if desired, be made lighter than water, the apparent sp. gr. varying, of course, with the proportions of the usual ingredients. The relation of compression strength to apparent sp. gr. is given by curves. The two diminish together, so that the lightest forms are only suitable for filling material etc. A further addition of the calcium alloy results in a part of the gas evolved being lost. With a proper mixture the gas pores are very uniform, and heat conductivity measurements show that the porous concrete is a very good insulating material. It can be structurally combined with iron in the same way as ordinary concrete. C. IRWIN.

Silica bricks. GROUM-GRJMAILLO. **Blast-furnace building materials.** DIEPSCHLAG and FEIST.—See VIII.

PATENTS.

Manufacture of acid-proof cements and acid-proof masonry. I. G. FARBERIND. A.-G. (B.P. 283,471, 9.1.28. Ger., 8.1.27. Addn. to B.P. 256,258; B., 1927, 333).—To acid-proof cements, prepared as previously described, may be added 2–4% of neutral material, other than silicon, silicon alloys, and fluosilicates, capable of reacting with alkalis to yield products more or less insoluble in acids, *e.g.*, tungstic acid, cryolite, or salts of fluozirconic, fluogermanic, hydrofluoric, fluotitanic, fluotantallic, fluoniobic, and fluostannic acids. L. A. COLES.

Manufacture of magnesia cements. Soc. "LE XYLUM" (B.P. 269,518, 2.4.27. Fr., 16.4.26).—The colloidal magnesium oxychloride formed in cements of this type is protected from weathering by the addition of an insoluble lead soap, as a waterproofing colloid, maintained in a state of dispersion by casein or a similar agent. A. COUSEN.

Manufacture of bricks and tiles. J. DUNLEVY and R. JOHNSON (B.P. 294,760, 5.7.27).—A mixture of glassmakers' waste sand, powdered limestone, and brickmakers' clay is worked to a plastic mass with water, moulded under pressure, and fired.

L. A. COLES.

Preservation of timber and other materials and destruction of insect and other pests therein. A. M. KOBIOLKE (B.P. 295,126, 9.8.27).—The timber is enclosed in an air-tight vessel which is successively evacuated, charged with impregnating liquor, drained to remove excess liquor when impregnation is complete, again evacuated, and suddenly filled with air by releasing the vacuum. L. A. COLES.

Hydraulic binding agents (B.P. 285,055).—See VII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Is the hearth chamber of a Siemens-Martin furnace a black body for optical pyrometry? H. SCHMIDT and W. LIESEGANG (Arch. Eisenhüttenw., 1927—8, 1, 677—685; Stahl u. Eisen, 1928, 48, 1049—1050).—The temperature of the walls of a Siemens-Martin, basic, open-hearth furnace has been measured through the peep-hole in the discharging door using a Wanner optical pyrometer with various coloured filters. The results showed a variation of up to 55° between the temperature measured through a red and that measured through a blue filter, indicating that the interior of the furnace does not radiate as a perfect black body. It would appear that the most reliable temperature measurements in such a furnace by means of an optical pyrometer should be obtained with a suitable blue filter instead of the red one usually employed. A. R. POWELL.

Change in elastic constant in metals caused by cold-working. K. HONDA and R. YAMADA (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 723—742).—The change in Young's modulus as the result of cold-working has been measured for iron, steel, copper, aluminium, and brass. For polycrystalline material the elastic constant is reduced by 6—10%, but may be largely recovered on annealing at a low temperature. Single crystals of iron showed a decrease of 3%, which was unaffected by annealing at 100°.

C. J. SMITHELLS.

Reactions between ferrous oxide and carbon and between carbon monoxide and iron. V. FALCKE (Z. Elektrochem., 1928, 34, 393—398; cf. A., 1926, 684; B., 1927, 191; Schenck, B., 1926, 633; A., 1927, 939, 1030).—Previous work on the equilibria $C + CO_2 \rightleftharpoons 2CO$ and $FeO + CO \rightleftharpoons Fe + CO_2$, chiefly by the author and by Schenck (*loc. cit.*), is summarised and discussed. Measurements have been made of the equilibrium constant of the first reaction, using as catalysts iron and nickel which had been previously heated in a stream of carbon monoxide. The values obtained with a nickel preparation containing 86.5% C are in agreement with those obtained with iron, whilst those with a nickel preparation containing less carbon are lower. The significance of this is discussed. The equilibrium constants of the reaction $C + CO_2 \rightleftharpoons 2CO$ were calculated by Schenck from the isochores of Jellinek and Diethelm (B., 1922, 972A). It is pointed out that these were incorrectly computed from the constants of amorphous carbon.

L. L. BIRCUMSHAW.

Equilibria in the system iron-carbon-oxygen. R. SCHENCK (Z. Elektrochem., 1928, 34, 399—403).—A résumé is given of previous work by the author and his collaborators on the equilibria in the reduction, oxidation, and carburization of iron, and a reply is made to the criticisms of Falcke (cf. preceding abstract). The line of future research in this field is indicated.

L. L. BIRCUMSHAW.

Repeated stress, structure, and damping [of special steels]. W. HEROLD (Arch. Eisenhüttenw., 1928—9, 2, 23—39; Stahl u. Eisen, 1928, 48, 1051—1052).—The structure of a pearlitic steel at the point of fracture by fatigue under repeated stress shows that the cementite has been forced towards the grain boundaries, whilst lamellar pearlite has been crushed and converted into granular pearlite giving the metal a very coarse-grained structure. In martensitic steels the hard η -phase becomes crushed into small particles which are forced through the softer ϵ -phase towards the grain boundaries. In some austenitic steels after fracture by fatigue, cementite may be observed at the grain boundaries, although the presence of this constituent is not visible in the original structure; in other cases the fatigued metal has a nodular structure which cannot further be resolved. Generally, steels which have been hardened and tempered lose their capacity for damping (*i.e.*, for converting energy into heat by internal friction) after subjection to repeated stress sufficient to produce failure by fatigue.

A. R. POWELL.

X-Ray investigation of density of quenched steels and internal stress existing within them. S. SEKITO (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 679—692).—The variation in the lattice parameter for a series of quenched carbon steels of different carbon contents has been measured, and found to be 0.45% for 1% C. This is in agreement with direct determinations of density, and indicates that the decrease in density obtained on quenching carbon steels results from an expansion of the lattice. From the broadening of the spectral line the calculated value of the internal stress is 192 kg./mm.², which is of the same order as the tensile strength. It is concluded that the increased strength results from the distortion of the iron lattice due to the presence of carbon atoms, and the distortion was found to be greatest in the direction perpendicular to that of easiest slip.

C. J. SMITHELLS.

Abrasion in carbon steels. M. SUZUKI (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 573—638).—The relative abrasion under a constant coefficient of friction has been measured for six hypoeutectoid steels having different carbon contents and heat-treatments. It is shown that if the specific abrasions of substances m and n with respect to a given substance N are W_{mN} and W_{nN} , then the relative abrasion $W_{mN} = \alpha \mu^2 W_{mN} / W_{nN}$, where μ is the coefficient of friction and α is a constant which remains invariable for a series of substances of similar properties, but varies when their properties differ considerably from one another. For hypoeutectoid steels martensitic, troostitic, sorbitic, annealed and rolled structures show decreasing abrasional resistance. With martensitic structures the minimum abrasion occurs with 0.7—0.8% C. C. J. SMITHELLS.

Determination of vanadium in steel. A. T. ETHERIDGE (Analyst, 1928, 53, 423–428).—Iron is removed from the steel as chloride by the ether extraction process, and other interfering metals by electrolysis in which a mercury cathode is used; the vanadium is then determined by the ordinary permanganate titration. The error from the titration is less than 0.01%. Slight modifications in the details of the procedure are given for high-silicon, tungsten, high-chromium, molybdenum, and high-manganese steels.

D. G. HEWER.

Effect of chromium on the electrical properties of iron-nickel alloys. P. CHEVENARD (Stahl u. Eisen, 1928, 48, 1045–1049).—Addition of chromium to nickel lowers the temperature of magnetic transformation very rapidly, the alloy with 6% Cr being paramagnetic at 20°. The anomaly in the resistance curve accompanying the magnetic change becomes feebler in intensity, and finally disappears with 4.35% Cr, whilst a new anomaly occurs in the paramagnetic state at 550° and increases in intensity with more than 2.35% Cr. The thermoelectric power of these alloys exhibits similar changes as the chromium content is increased, but with 8–10% Cr the alloys have a high positive thermoelectric power which is practically free from irregularities. In iron-nickel alloys with more than 50% Ni addition of chromium produces similar effects on the electrical properties, and in alloys with low percentages of nickel it lowers the temperature of the Ar₂ and Ar₃ points and enlarges the region of the reversible austenitic alloys towards the iron side.

A. R. POWELL.

Variation of the composition of alloys of silver and copper melted in contact with air. GUICHARD, CLAUSMANN, and BILLON (Bull. Soc. chim., 1928, [iv], 43, 752).—The variation is due to the contrary effects of the disappearance of copper by progressive oxidation, and to volatilisation which affects the silver more than the copper. It therefore depends on the temperature, the silver content rising at 1000° and falling at 1300°, whilst at 1100° equal amounts of silver and of copper are removed and the composition remains constant.

J. GRANT.

Deoxidation of silver castings. GUICHARD, CLAUSMANN, and BILLON (Bull. Soc. chim., 1928, [iv], 43, 752–753).—Instead of a protecting layer of powdered carbon, about 0.001 pt. of copper phosphide added to silver-copper castings acts as an efficient deoxidant. A smaller number of reheatings for hammering-out are required.

J. GRANT.

Laboratory experiments on high-temperature resistance alloys. C. J. SMITHELLS, S. V. WILLIAMS, and J. W. AVERY (Inst. Metals, Sept., 1928. Advance copy. 22 pp.).—Resistance to oxidation at high temperature was studied by electrically heating a helix of standard dimensions, formed from an alloy wire, and observing the endurance or life of the wire under the particular conditions which were comparable with the use of the alloy in practice. Thus a helix of nickel-chromium wire was attached to the lead-in wires of an electric lighting bulb. The influence of adventitious draughts when the

helix was heated in the open air was less than when enclosed in a glass bulb, as the loss of chromium as sodium chromate outweighed any increase in weight due to oxidation. For the binary alloys of nickel and chromium, resistance to oxidation increased as the chromium content increased up to 30%. A second phase appeared with more than 40% Cr and the alloy oxidised more readily. Ternary alloys containing 10% Cr were low in resistance, which, however, was high when 20% Cr was present. Whilst the resistance to oxidation is due to the formation of an oxide coating, the composition of the oxide is determined by, but not necessarily the same as, the composition of the alloy, and the oxide must contain at least 50% Cr₂O₃ to render adequate protection against further oxidation. Resistance to sag of similar wires determined by deformation of a wire of hairpin shape when heated showed that increasing chromium content increased the tendency to sag; a 50% Cr alloy sagged completely at 1050° and 900°, but was rigid at 750°. Ternary alloys sagged slightly more than binary alloys having similar contents of nickel, and alloys made from prepared pure metals were considerably more resistant than commercial alloys containing usual impurities. Electrical resistance curves showed that at room temperature resistance increases with decreasing nickel. Substitution of chromium by tungsten lowers resistance, whilst molybdenum has the opposite effect. Generally, maximum resistance was shown at 500–550°.

C. A. KING.

Strength of a cadmium-zinc and of a tin-lead alloy solder. C. H. M. JENKINS (Inst. Metals, Sept., 1928. Advance copy. 19 pp.).—With reference to use for soldering purposes comparative strengths of the eutectic cadmium-zinc and 60:40 tin-lead alloys were determined. Prepared in a similar manner the strength of the cadmium alloy was 3–4 times as great as for the tin solder, the cast alloy with a value of about 10 tons/in.² being by far the strongest. Cold-rolled material possessed a decidedly lower tensile strength, a value as low as 1.7 tons/in.² being found for the tin-lead alloy; similar low values were also shown by aged (120°) and long-annealed alloys. None of the cadmium-zinc specimens showed intercrystalline cracking, and under prolonged loading and heat-treatment conditions this alloy showed greater relative strength than the tin alloy.

C. A. KING.

Treatment of aluminium and aluminium alloys with chlorine. D. R. TULLIS (Inst. Metals, Sept., 1928. Advance copy. 8 pp.).—In order to secure effective degasification of aluminium and its alloys the necessity is assumed for introducing an element which disturbs the equilibrium existing between aluminium and the dissolved gases, e.g., magnesium, zinc. The removal of dissolved gases was attempted by passing a highly active gas (chlorine) through the molten alloy. Considerable increase in density was observed in the gassed alloys and general improvement in the castings; large surface crystals were evident with cast silicon-aluminium alloys when gassed, and eutectic extrusion was less than when slowly solidified. Aluminium alloys containing 10% Fe were responsive to chlorine treatment, but with 15 and 20% Fe it appeared to be necessary to add an element which causes gas disturbance.

C. A. KING.

"Rockwell" hardness test [for metals]. J. E. MALAM (Inst. Metals, Sept., 1928. Advance copy. 20 pp.).—Data obtained to attempt to correlate the hardness of metals, in particular copper-zinc alloys, by Brinell, Rockwell, and scleroscope methods showed that the Rockwell number is comparable with the Brinell number only for certain critical values, and, generally, the Rockwell ball-test must be considered misleading. Certain modifications in the method of expressing the results of the Brinell and scleroscope tests are desirable.

C. A. KING.

Determination of "objectionable" sulphur in roasted [zinc] blende. E. BEYNE (Ann. Chim. analyt., 1928, [ii], 10, 221—225).—The sulphur content of roasted blends may occur as undecomposed sulphides, as zinc sulphate, or as lead, magnesium, barium, or calcium sulphate. None of the four last-named sulphates can be further decomposed by roasting, hence the term "objectionable" sulphur is applied only to that existing as sulphide or as zinc sulphate. The latter is determined by extracting the material with hot water and determining the soluble zinc, and the former by the evolution method (boiling with hydrochloric acid and stannous chloride and oxidising the hydrogen sulphide evolved).

A. R. POWELL.

Pyrometry. BROOK and SIMCOX. **Densities of ore pulps.** FAHRENWALD.—See I. **Blast-furnace firebricks.** DIEPSCHLAG and FEIST.—See VIII. **Testing magnet steel.** BABBITT.—See XI.

PATENTS.

Cupola furnaces. FREIER GRUNDER EISEN- U. METALLWERKE GES.M.B.H. (B.P. 294,027, 13.10.27. Ger., 21.7.27. Addn. to B.P. 267,008).—To assist discharge of the residue of slag and iron from a cupola, the downwardly inclined passage from the slag separator is arranged at the lowest point of the base of the furnace shaft. A closed lower tapping hole is connected in line with the separator to allow removal of residues.

C. A. KING.

Metallurgical furnace. F. B. McKUNE, Assr. to OPEN HEARTH COMBUSTION Co. (U.S.P. 1,679,340, 31.7.28. Appl., 5.5.22).—A reversible metallurgical furnace is provided with air entrance passages, means for restricting the admission of air to the furnace chamber, and fans for supplying forced air to the passages.

M. E. NOTTAGE.

Metallurgical separator. A. T. SWEET, Assr. to W. G. RICE, H. GUNDLACH, and P. B. WOODWORTH (U.S.P. 1,678,884, 31.7.28. Appl., 21.10.26).—The ore is caused to move in a stream across a beam of light, separation of the ore being effected owing to variations of light-reflecting values between the minerals and the remainder of the stream.

M. E. NOTTAGE.

Enriching oxide iron ores, flue dust from blast furnaces, burnt pyrites, purple ore, etc. AKTIEBOLAGET FERRICONCENTRAT, Assees. of H. A. MUELLER (B.P. 279,797, 3.9.27. Swed., 28.10.26).—Weakly paramagnetic ores are heated in an oxygen-free atmosphere to a strongly paramagnetic condition and then separated by means of a low-intensity magnetic separator. Carbonate ores are given a preliminary roasting in air.

C. A. KING.

Manufacture of stainless iron. A. L. FEILD (B.P. 282,387, 10.12.27. U.S., 18.12.26).—Stainless iron within the usual limits of composition is produced from a bath of molten iron, a material high in iron oxide, and an iron-chromium alloy relatively high in carbon. The operation is conducted so that chromium is oxidised and enters the slag and the metal is decarbonised. The chromium content of the metal is then increased by introducing a non-carbonaceous reducing agent, e.g., silicon, ferrosilicon, and final adjustment may be made by adding stainless iron or steel scrap.

C. A. KING.

Flux for welding cast iron. C. C. MARYAN, Assr. to GEN. ELECTRIC Co. (U.S.P. 1,676,988, 10.7.28. Appl., 8.12.25).—The flux contains 4 pts. of ferric carbonate, 12 pts. of ammonium chloride, 16 pts. of sodium borate, 20 pts. of sodium bicarbonate, and 12 pts. of sodium chloride.

H. ROYAL-DAWSON.

Copper-chromium ferrous alloys. B. D. SAKLATWALLA (U.S.P. 1,676,929, 10.7.28. Appl., 7.4.24).—Iron containing carbon is alloyed with 8—25% Cr, 0.5—5% Cu, and 1—3% Si.

H. ROYAL-DAWSON.

Thermostatic material. V. G. VAUGHAN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,678,889; 31.7.28. Appl., 12.9.25).—In the thermostat one element consists of an alloy of cobalt and iron, the other of nickel-steel.

H. ROYAL-DAWSON.

Solvent treatment of copper ores. ANGLO-AMER. CORP. OF S. AFRICA, LTD. (B.P. 294,868, 22.5.28. S. Africa, 7.12.27).—The cupriferous material is reduced by wet grinding to a slime pulp which is then dewatered by filtration, compressed air being subsequently forced through the resulting cake. This pulp is then treated with a suitable ammoniacal solvent (e.g., a cupric ammonium carbonate solution), the cake being re-pulped with the solvent solution and again filtered, or, preferably, the dewatered pulp is formed as a cake in a pressure filter and the solvent solution forced through it.

M. E. NOTTAGE.

Bearing-metal alloys. H. and S. BONDI and O. NEURATH [J. NEURATH] (B.P. 283,862, 16.1.28. Poland, 17.1.27. Addn. to B.P. 238,895; B., 1926, 62).—0.5—3.5% of indium or cadmium is added to the alloy previously described.

H. ROYAL-DAWSON.

Extraction of zinc. A. ROITZHEIM and W. REMY (B.P. 294,127, 6.4.27).—In a zinc furnace of which the heating is aided by a partial combustion of carbon in the furnace, volatilised zinc oxide is condensed and recovered in baffled chambers at 900—1000°, whilst zinc vapours pass into a further chamber and are condensed at 850—500°.

C. A. KING.

Aluminium alloys. DEUTS. VERSUCHSANSTALT F. LUFTFAHRT E.V. (B.P. 282,701, 6.4.27. Ger., 27.12.26).—Aluminium alloys containing copper and magnesium (in which both the Mg_2Si and $CuAl_2$ can exert their full effect during ageing of duralumin at room temperature) and having improved mechanical properties may be made from aluminium electrolytically purified or chemically free from iron, the necessary amount of silicon for the formation of $MgSi_2$ being added in the form of aluminium-silicon.

M. E. NOTTAGE.

Manufacture of alloys of nickel and chromium. GEN. ELECTRIC Co., LTD., and C. J. SMITHELLS (B.P. 293,727, 7.4.27).—The alloys consist either of nickel and chromium in which the chromium content is 10–50%, or of nickel and chromium together with molybdenum and/or tungsten, in which the chromium content is above 10% and the nickel above 60%. The pure metals, prepared electrolytically in the form of powder by the use of a current of high density, are consolidated in an atmosphere of hydrogen either by sintering at a temperature below their m.p., preferably with intermediate working, or by fusion, in which case the metal powders may be heated either by an outside furnace or by means of an induced current. In the latter case the enclosure containing the hydrogen may be cooled.

M. E. NOTTAGE.

Manufacture of tools of tungsten. O. DIENER (B.P. 294,086, 15.2.28).—Powdered carbonaceous tungsten, containing more than 3% of combined carbon and no auxiliary metals, is heated at about 2000°, and a reaction pressure of about 150–200 kg./cm.² is simultaneously applied, so that separation of the combined carbon is prevented. Up to 0.6% B or 1.5% Si, or 1.5% (B + Si), may be added to facilitate the process of pressing and still further to increase the hardness.

M. E. NOTTAGE.

Concentration of mixed sulphide ores. H. S. MARTIN (U.S.P. 1,678,259, 24.7.28. Appl., 30.6.27).—A substance which, under the conditions of the flotation process, is capable of liberating the sulphite radical and hydrogen ions is added to the pulp (made alkaline), in order to effect the selective froth-flotation of copper sulphides from ores containing iron sulphides.

H. ROYAL-DAWSON.

Flotation method [for ores]. G. L. ADAMS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,678,311, 24.7.28. Appl., 30.1.24).—The flotation agent claimed in U.S.P. 1,678,312 (B., 1928, 702) is used for the concentration of ores.

H. ROYAL-DAWSON.

Manufacture of electrical resistance elements. GEN. ELECTRIC Co., LTD., and C. J. SMITHELLS (B.P. 293,728 and 293,740, 7.4.27).—(A) Nickel-chromium alloys free from deleterious impurities are claimed, the content of nickel being above 50% and of chromium above 10%. In an example the alloy contains 70% Ni and 30% Cr. (B) Resistance to oxidation of such alloys may be increased by substituting molybdenum or tungsten for part of the chromium. Suitable alloys contain 70% Ni, 20% Cr, and 10% W or Mo.

M. E. NOTTAGE.

Blast furnaces [for iron ores]. BROKEN HILL PROPRIETARY Co., LTD. (B.P. 282,042, 1.12.27. Austral., 9.12.26).—See U.S.P. 1,664,832; B., 1928, 411.

Heat-treatment and concentration of copper ores. J. C. MOULDEN and B. TAPLIN, Assrs. to METALS PRODUCTION Co. OF N. AMERICA, INC. (U.S.P. 1,679,337, 31.7.28. Appl., 14.9.25. U.K., 29.10.24).—See B.P. 250,991; B., 1926, 590.

Treatment of ores of copper and other metals. F. DIETZSCH (U.S.P. 1,679,294, 31.7.28. Appl., 31.10.27. U.K., 7.7.26).—See B.P. 281,741; B., 1928, 128.

Welding electrodes [for nickel]. N. B. PILLING and J. G. SCHOENER, Assrs. to INTERNAT. NICKEL Co. (U.S.P. 1,679,002, 31.7.28. Appl., 19.10.25).—See B.P. 259,967; B., 1927, 784.

[Torch for] surface-hardening of metal articles. G. H. FLETCHER, C. L. SUMPTER, and METROPOLITAN-VICKERS ELECTRICAL Co., LTD. (B.P. 294,709, 3.5.27).

Metallurgical coke (U.S.P. 1,676,729).—See II. **Furnace electrodes** (B.P. 282,672).—See XI.

XI.—ELECTROTECHNICS.

Permeameter for testing magnet steel. B. J. BABBITT (J. Opt. Soc. Amer., 1928, 17, 47–58).—The common commercial permeameters are not capable of producing the high magnetising forces required to determine accurately the magnetic properties of cobalt steel in bar form. The yoke type of permeameter may be adapted for this purpose by the use of extensions to the poles so that the distance between them is much less. In this way the greater part of the magnetomotive force is distributed over a short portion of the magnetic circuit and the force per cm. is correspondingly greater. The instrument described gives a very uniform magnetising force, directly proportional to the magnetising current, throughout a wide range of values. It is automatically compensated so that it records only ferric induction, is simple and rapid in operation, and possesses a high degree of accuracy.

L. L. BIRCUMSHAW.

Coal conductivity cell. SINKINSON.—See II. **Iron-nickel alloys.** CHEVENARD. **Resistance alloys.** SMITHELLS and others.—See X. **Penetrability of leather.** GERSSEN.—See XV.

PATENTS.

Ironless induction furnaces or heating apparatus. SIEMENS & HALSKE A.-G. (B.P. 277,352 and 277,361, [A] 8.9.27, [B] 9.9.27. Ger., [A, B] 11.9.26. Addns. to B.P. 274,888; B., 1928, 415).—(A) The induction coil of the furnace is enclosed in a double-walled cylinder so that the coil is protected, and supported, and has also free movement for expansion particularly in the direction of the axis of the coil. The protective cylinder may be of carborundum or other suitable refractory. (B) The induction coil is inserted from the bottom of the furnace so that the outer and inner lines of force of the coil pass through the charge. In one arrangement hollow refractory cylinders are provided in the base of the furnace and are accessible from the outside.

C. A. KING.

Furnace electrodes [for manufacture of aluminium]. SOC. ELECTRO-MÉTALLURGIQUE DE MONTRICHER (B.P. 282,672, 17.12.27. Fr., 21.12.26).—To prevent overheating of the electrodes each is formed of sections dove-tailed and bolted together as a cylindrical casing which is filled with a non-conducting material. The upper part of the conducting casing is mounted on metal rings and bars for the better distribution of the current.

C. A. KING.

Furnace electrodes. SOC. ELECTRO-MÉTALLURGIQUE DE MONTRICHER (B.P. 294,837, 20.12.27. Fr., 13.10.27. Addn. to B.P. 282,672; preceding).—The

longitudinal metal bars in the electrodes previously described comprise vertical stanchions in contact over their whole length with the inner faces of the conducting sections, and attached to means for raising and lowering the electrode. Adjacent sections of the electrode are connected together by a paste of carbonised sugar or glucose under pressure. L. A. COLES.

[Filling for] gas-filled electric lamps. SIEMENS ELECTRIC LAMPS AND SUPPLIES, LTD., and P. D. OAKLEY (B.P. 295,072, 13.5.27).—A filling for gas-filled lamps having a filament composed wholly or mainly of tungsten consists of a mixture of an inert gas or gases, *e.g.*, a mixture containing 85% of argon and 15% of nitrogen together with 0.2—1% of a chlorine-substitution product of an aliphatic hydrocarbon, *e.g.*, ethyl chloride. J. S. G. THOMAS.

[Filling for] electric incandescence lamps with an incandescent body of tantalum carbide. B. ERBER (B.P. 286,687, 16.2.28. Austr., 9.3.27).—The lamps are filled with an inert gas, *e.g.*, argon, to prevent decomposition of the carbide. J. S. G. THOMAS.

Obtaining a perfect high vacuum in electric discharge tubes. S. LOEWE (B.P. 279,842, 24.10.27. Ger., 26.10.26).—In the assembling of electric discharge tubes, a plate of magnesium is situated at a sufficient distance from the anode as to be heated separately by high-frequency eddy currents. During the process of evacuation the anode is heated first for the discharge of occluded gases, then by raising the inductance coil the magnesium is heated to dull redness before heating to a higher temperature to disperse the metal. C. A. KING.

Obtaining a very high vacuum [in electric discharge tubes]. S. LOEWE (B.P. 280,908, 14.11.27. Ger., 16.11.26).—The residual traces of gas are bound by a double dispersion of magnesium metal by high-frequency induction before and after sealing off. The second dispersal is effected at a point in the bulb remote from the first so that the first magnesium mirror should not be affected by the second heating. As an additional precaution the second dispersal may be effected in a space between mica discs. B. M. VENABLES.

Introduction of chemically active [alkali] metals into evacuated or gas-filled containers. WESTINGHOUSE LAMP CO., Assecs. of J. W. MARDEN (B.P. 267,902, 23.2.27. U.S., 20.3.26).—A mixture of a non-hygroscopic, anhydrous compound of potassium, rubidium, caesium, or barium, *e.g.*, a chromate, dichromate, permanganate, or double halide (potassium zirconium fluoride etc.), with a reducing agent, *e.g.*, misch metal, iron, nickel, aluminium, or magnesium, preferably enclosed in a metal capsule of low m.p., is decomposed inside the container by heating. L. A. COLES.

Production of metal-containing electric resistances. M. HAUSER (B.P. 270,301, 26.4.27. Switz., 28.4.26).—The resistances comprise mixtures of silicon or ferrosilicon with a high silicon content, a chromium alloy, *e.g.*, ferrochromium, boric acid or a borate, and ceramic raw material, the mixtures being fired under such conditions that oxidation of the charge is avoided. L. A. COLES.

Manufacture of accumulator plates. I. G. FARBERIND. A.-G. (B.P. 284,352, 28.1.28. Ger., 28.1.27).—A lead grating is cast around a casting of an alloy of lead with a metal, *e.g.*, sodium, which is subsequently removed by dissolution. J. S. G. THOMAS.

Art of galvanoplasty. UNITED PRODUCTS CORP. OF AMERICA (B.P. 273,664, 29.4.27. U.S., 3.7.26).—The preparation of a non-metallic article for the electro-deposition of a metal consists in coating the article with a colloidal film of rubber, making the film conductive, and then rendering the film water-resistant. Thus a film of colloidal rubber containing sulphur may be partially dehydrated, coated with powdered metal when "tacky," vulcanised, and coated with metal by electro-deposition. C. A. KING.

Heat-sensitive recording papers and the like suitable for use in picture and the like telegraphy. MARCONI'S WIRELESS TELEGRAPH CO., LTD., Assecs. of R. H. RANGER (B.P. 276,020, 13.8.27. U.S., 14.8.26).—Paper suitable for recording with a jet of hot air or an electric spark is coated with a composition comprising, *e.g.*, 5 g. of nickel nitrate, 3.9 g. of sodium thiosulphate, 3.6 g. of sodium nitrate, and 5 c.c. of 1% gelatin solution per 100 c.c. of water. L. A. COLES.

Electric bright-annealing furnaces. W. ROHN, ASSR. to SIEMENS-SCHUCKERTWERKE GES.M.B.H. (U.S.P. 1,678,875—6, 31.7.28. Appl., [A, B] 23.10.25. Ger., [A] 15.12.24, [B] 1.11.24).—See B.P. 244,425 and 242,283; B., 1927, 115; 1926, 834.

Flexible [laminated] insulating material. BRIT. THOMSON-HOUSTON CO., LTD., Assecs. of L. V. ADAMS (B.P. 280,189, 25.10.27. U.S., 8.11.26).

Resistance elements (B.P. 293,728 and 293,740).—See X. Rubber (B.P. 293,815).—See XIV.

XII.—FATS; OILS; WAXES.

Analysis of glycerin by the acetin method, and its sources of error. O. BERTH (Chem.-Ztg., 1928, 52, 597—598, 619—620).—The international standard acetin method for the analysis of commercial glycerin yields slightly (0.2—0.3%) high results owing to the presence of carbon dioxide in the alkali used and to absorption of that gas from the air. To obviate these errors the standardisation of the alkali against the acid should be carried out under exactly the same conditions as the analysis, except that no glycerin is used, *i.e.*, the acetic anhydride and sodium acetate are boiled together with water, the solution is neutralised to phenolphthalein with sodium hydroxide, a further 50 c.c. are added in excess, and, finally, the mixture is titrated with *N*-hydrochloric acid. This value is used in calculating the results. A. R. POWELL.

Chemistry of lead "soaps" made from litharge and neutral fat. S. H. DIGGS and F. S. CAMPBELL (Ind. Eng. Chem., 1928, 20, 828—829).—When fats are "saponified" with lead oxide in the absence of water at 149°, no free glycerol is formed, nor is any appreciable water or aldehyde liberated. The reaction appears to be one of addition. It can only be carried out satisfactorily if excess of fat or some other diluent such as paraffin oil is present in order to facilitate thorough

mixing during the heating. Unlike the normal and basic fatty acid salts of lead made from lead oxide and fatty acid, these "soaps" from neutral fats show no indication of a colloidal nature, nor are their molecules associated in solution. F. R. ENNOS.

Simpler derivation of the Cook formula for determination of acetyl value of fats and oils. W. RIEMANN III and A. T. HAWKINSON (J. Amer. Chem. Soc., 1928, 50, 2311—2312).—Cook's formula (B., 1922, 299 A) is derived by a simple calculation.

R. K. CALLOW.

Drying oils. VIII. Adsorption of liquids by oil gels. J. S. LONG, E. K. ZIMMERMANN, and S. C. NEVINS (Ind. Eng. Chem., 1928, 20, 806—809). **IX. Action of cold-blowing on linseed oil.** J. S. LONG and W. S. EGGE (*Ibid.*, 809—811).—VIII. The adsorption of various fatty acids, raw linseed oil, mineral oil, glycerol, etc. by extracted heat-bodied oil gels and extracted linseed oil films was measured. The results indicate that the semi-solid material obtained by heat-bodilying of drying oils actually are gels, and that films made by the ordinary drying process have in part a similar gel structure. The gels of both types show similar adsorptions, are equally softened by fatty acids, and are converted into similar tough, rubbery products by the adsorption of mineral oil and glycerol. The heat of combustion is put forward as a criterion for studying the progress of oil-oxidation, the value decreasing from 9367 g.-cal. for raw linseed oil to about 7000 g.-cal. for dry linseed oil films. The heats of combustion of solid and liquid phases in oil films of increasing ages were determined, the value for the solid phase (after extraction) remaining sensibly constant, whilst that for the extracted oil decreased steadily from the much higher value characteristic of slightly oxidised oil. It is considered that the adsorption of the liquid phase by the solid gel structure in the course of solidification of oil involves definite orientation as opposed to simple mechanical entanglement. The oriented, adsorbed, liquid phase changes chemically into the complex molecules characteristic of the solid.

IX. Linseed oil was blown at 30° in the presence of manganese driers, α -naphthol, and sodium and calcium oleate, and after 8 hrs. heating in an inert atmosphere at various temperatures. The changes in d , n_D^{20} , acid value, iodine value, and hexabromide value are recorded. The decrease in unsaturation observed in the presence of positive catalysts is inhibited in the presence of negative catalysts (even if positive catalysts be also present). The oleates lower the surface tension of the oil and permit the immediate formation of a thick, stable foam, the rapid oxygen-absorption at the thus increased interface causing a given viscosity to be attained in about half the time taken by a positively catalysed oil. Previous heat-treatment also proportionately shortens the time taken to reach a given "body." The addition of 0.5% of thioglycollic acid was found quantitatively to precipitate the metals of metallic driers in oils and varnishes. Apart from its analytical significance, this may be used to study the course of particular reactions, driers being withdrawn from the system at any desired stage. S. S. WOOLF.

[Criticism of] a colorimetric reaction to determine the purity of olive oil. M. CUCCURULLO (Annali Chim. Appl., 1928, 18, 297—299).—The eosin test described by Milani (B., 1927, 915; 1928, 130), and apparently dependent on the amount of free acid present, is unreliable; pure olive oil may give a permanent coloration. E. W. WIGNALL.

Laboratory apparatus for extraction of oils with solvents. H. V. DE CAMPOS (Chimica e Ind., 1928, 3, 482—484).—Descriptions and diagrams are given of apparatus, which may be constructed from ordinary laboratory materials, for the extraction of oil from seeds etc. in quantities of up to 5 kg., by either hot or cold solvents, working on the Soxhlet principle.

R. K. CALLOW.

Testing oleine oils by the thiocyanate method. H. P. KAUFMANN (Z. angew. Chem., 1928, 41, 19—22).—The results of examination of a number of samples of oleine obtained from various Continental manufacturers, and used for lubricating textile fibres during processes of manufacture, indicate that the susceptibility to oxidation and consequent spontaneous ignition of an oleine is proportional to the difference between its iodine and thiocyanate values. A. J. HALL.

Locust-kernel oil. WILLIAMS.—See XVII. **Cod-liver meals.** BETHKE and others.—See XIX.

PATENTS.

Treatment of oils or fats or mixtures of the same or fatty acids for the production of sulphuric acid compounds. E. C. R. MARKS. From CHEM. FABR. STOCKHAUSEN & CO. (B.P. 293,717, 6.4.27).—Sulphonated products of great stability are obtained by treating oils etc. with concentrated sulphuric acid, in excess of 35%, at temperatures not above 10—15° (cf. B.P. 293,480; B., 1928, 678). Addition of the acid should be as rapid as possible to prevent excessive rise in temperature. E. LEWKOWITSCH.

Extraction and preparation of animal fats rich in vitamins. J. LYONS & Co., LTD., L. H. LAMPITT, and J. H. BUSHILL (B.P. 293,777, 5.4.27).—Visceral material is frozen, comminuted, thawed, and treated with caustic soda at 40° to dissolve protein matter. The fat is neutralised and separated. E. LEWKOWITSCH.

Obtaining certain remedial principles of oils. T. F. ZUCKER, ASSR. to UNIVERSITY PATENTS, INC. (U.S.P. 1,678,454, 24.7.28. Appl., 7.12.22).—Cod-liver oil is extracted with alcohol, the fatty acids in the extract are saponified, and the soaps converted into calcium soaps, from which the antirachitic principles are extracted.

B. FULLMAN.

Dye soaps. W. KRITCHEVSKY and H. C. PRUTSMAN (B.P. 270,637, 29.12.26. U.S., 5.5.26).—Dye soaps which give no scum when used with hard water and do not dry out on storage nor liberate fatty acid when prepared with acid dyes are made from sulphonated soaps of the general formula $\text{CO}_2\text{X} \cdot \text{R} \cdot \text{O} \cdot \text{SO}_3\text{X}$, where X represents a metal and R a hydrocarbon radical or a derivative thereof. [Stat. ref.] D. J. NORMAN.

Bleaching of fats (B.P. 274,828).—See II. **Phosphatides** (U.S.P. 1,673,615).—See XIX.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Chipping and abrasion tests for paint coatings on metal. A. D. CAMP (Ind. Eng. Chem., 1928, 20, 851—852).—The chipping test is made by dropping a series of tools of specified dimensions down a 24-in. tube on to standardised, painted, test panels and observing the extent and character of the impression made on the coating. The abrasion test is made by determining the number of revolutions of the painted test panel under a standard abrading disc which are required to wear through the paint film. S. S. WOOLF.

Yellowing of white enamels. H. MUNZERT (Farben-Ztg., 1928, 33, 2849—2851).—Previous German work on the yellowing of enamel vehicles with age is summarised. A series of comparable enamels were prepared, using equal bulks of zinc sulphide ("Sachtolith"), titania, zinc oxide, and white lead, all in a high state of purity, with each of the following vehicles (white spirit being the solvent):—60% solution of lime-hardened rosin, 60% solution of ester gum, 70% solution of pale linseed oil stand oil, and 60% solution of a tung oil-linseed oil stand oil. The 16 enamels were brushed on tin plate, and dried for 4 days in diffused daylight. The films were then half-covered by black paper and exposed to direct sunlight for 14 days, after which the degree of yellowing was noted in comparison with freshly-prepared films of the stored enamels and with films of similar freshly-prepared enamels. The covered films were found to yellow more than the exposed ones, whilst of the pigments white lead caused most yellowing, followed by zinc oxide, titania, and zinc sulphide in descending order. Yellowing is attributed in the main to dark-coloured oxidised acids which rapidly undergo further degradation in the light to form gaseous or colourless compounds. This degradation is, however, delayed in the dark and to a greater degree still by salt-formation with pigments, the "fixing" of the yellowing being proportional to the basic nature of the pigment.

S. S. WOOLF.

Graphical methods in lacquer technology. H. E. HOFMANN and E. W. REID (Ind. Eng. Chem., 1928, 20, 431—436).—Rectangular co-ordinates are useful in interpreting the results of distillation and evaporation tests of solvents, and tests of tensile strength and elongation under load, of lacquer films. The values of dilution ratios when a standard non-solvent is added to a solution of nitrocellulose in a true solvent until precipitation begins may be represented by bars or lines parallel to the ordinate. For two non-solvents mixed in varying proportions the dilution ratio is plotted against the composition of the diluent mixture. The use of triangular co-ordinates in the investigation of the capacity of a solvent mixture of three components to dissolve nitrocellulose is described. Constant-cost mixtures may also be similarly represented. Examples are also given of the application of nomographs in mixing lacquers to give a product of desired viscosity or weight per gallon.

W. J. BOYD.

Phenol-formaldehyde resinification. I. J. NOVÁK and V. ČECH (Ind. Eng. Chem., 1928, 20, 796—801).—The isolation and purification of phenol-aldehyde condensation products at intermediate stages of the resini-

fication having proved impracticable, the authors have studied the kinetics of the reaction by observing changes in refractive index, viscosity, and "bromine value." The methods adopted are described fully. Typical resinifications in aqueous mixtures were examined by these methods without catalyst and with alkaline and acid catalysts. The progress and products of resinification are shown to differ considerably in each case, and the reaction mechanisms are discussed. In the absence of catalysts condensation reactions occur until an intermediate insoluble resinous substance is precipitated; this now undergoes polymerisation, but the condensation also continues. With alkaline catalysts the initial condensation is accelerated and the subsequent polymerisation is intensified. Products of acid resinification, however, have a very limited tendency to polymerisation. S. S. WOOLF.

Drying oils. LONG and others.—See XII.

PATENTS.

Non-livering coating compositions. E. I. DU PONT DE NEMOURS & Co., Assecs. of J. D. MCBURNEY and E. H. NOLLAU (B.P. 294,029, 20.10.27. U.S., 22.7.26).—Livering of coating compositions with a drying-oil base is prevented or removed by the admixture of small quantities (at least 0.1%) of substances with a dissociation constant between 1 and 3.4 (*e.g.*, citric acid, tartaric acid). E. LEWKOWITSCH.

Coating and binding compositions. C. TAUBER [G. LEUCHTE NACHF.] (B.P. 275,610, 27.7.27. Ger., 3.8.26).—Vehicles for primers for wood, brick, plaster, etc. are obtained by incorporating magnesium soaps with linseed oil, tung oil, etc. The stronger basic character of magnesia compared with alumina (as previously claimed) obviates reaction of the soaps with basic pigments in the mixed paint. [Stat. ref.] S. S. WOOLF.

Production of permanent paste containing red oxide of lead (minium). E. ASSER (B.P. 294,436, 9.1.28).—Red lead is ground in linseed oil, or other paint vehicle, in the presence of a small proportion of infusorial earth and/or a neutral or basic resinic or fatty acid salt of alumina that has undergone colloidal swelling in a suitable solvent, *e.g.*, benzene. The paste thus produced does not harden, and may be converted into paint by the addition of linseed oil etc.

S. S. WOOLF.

Production of cellulose varnishes. H. WOLFF and R. SINGER (B.P. 293,732, 11.4.27).—Stable cellulose lacquers containing lead pigments are obtained by the introduction of acid organic compounds (*e.g.*, picric acid, fatty acids, etc.). E. LEWKOWITSCH.

Production of coloured brushwork lacquers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 294,158, 19.4.27).—Coloured cellulose derivatives prepared from nitrocellulose, acetylcellulose, or water-insoluble cellulose ethers (cf. B.P. 247,238 and 293,435; B., 1926, 315; 1928, 668) are dissolved in glycol mono- or di-alkyl ethers or their esters or other similar solvents, and the resulting lacquers are diluted with aromatic hydrocarbons or their halogen derivatives and/or aliphatic or hydroaromatic alcohols. The products are

brushable and give coatings of high gloss without further treatment when dry. S. S. WOOLF.

Making resinous compositions. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of E. S. DAWSON, JUN. (B.P. 285,459, 15.2.28. U.S., 17.2.27).—Drying oil is compounded with a "glyptal" resin (cf. B.P. 235,595; B., 1926, 638) by dissolving the oil in the polybasic organic acid component, *e.g.*, phthalic anhydride, and reacting on this solution with the polyhydric alcohol component, *e.g.*, glycerol, at a temperature sufficiently high to produce resinification, no separate solvent being used. Alternatively, the three components may be treated together. S. S. WOOLF.

Manufacture of phenol-formaldehyde condensation products. KUNSTHARZFABR. DR. F. POLLAK GES.M.B.H. (B.P. 267,901, 23.2.27. Austr., 17.3.26).—A colloiddally dispersed condensation product of 1 mol. of phenol and $2\frac{1}{2}$ mols. of formaldehyde is produced by conducting the main condensation under strongly alkaline conditions. The colloidal solution of the intermediate product is neutralised and gelled by heat. The gel is finally hardened by heat, preferably under weakly acid conditions (the use of halogenated lower fatty acids being recommended for the neutralisation process). S. S. WOOLF.

Manufacture of a powder which can be pressed by the condensation of urea or its derivatives and formaldehyde. KUNSTHARZFABR. DR. F. POLLAK GES.M.B.H. (B.P. 271,037, 28.1.27. Austr., 15.5.26).—An aqueous solution of the condensation product is diluted to such an extent, or, alternatively, the condensation takes place at such a dilution, that no gelatinisation takes place, and the product is precipitated as an amorphous, flocculent precipitate by the addition to the solution of a flocculating agent, *e.g.*, an acid, base, or albumin precipitant. The precipitate is washed, dried, and moulded under heat and pressure into glass-clear objects. L. A. COLES.

Plastic [insulating] material and its manufacture. P. E. BASSET (B.P. 293,436, 21.11.27. Fr., 8.7.27).—Vegetable proteic materials and glutens (*e.g.*, spent grains, malt, etc.) are condensed with phenolic compounds under the influence of catalysts or hydrolysing substances. E. LEWKOWITSCH.

Arylaminesulphonic acid derivatives (B.P. 293,781).—See III. **Azo dyes** (B.P. 294,291). **Insoluble colouring materials** (B.P. 270,293).—See IV. **Fluorescent preparations** (B.P. 295,078).—See VII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Dispersoidological investigations on latex. P. P. VON WEIMARN (Bull. Chem. Soc. Japan, 1928, 3, 157—168).—A *résumé* of the salient features of recent investigations, details of which will appear later. Amongst the microscopically visible particles are particles of all the shapes reported by previous observers. The main conclusion regarding the structure of latex is expressed as follows: Latex is a polydisperse system of iso-aggregate particles (iso-spherulites), the general consistency of which is fluid-gelatinous. The individual solid particles which compose the iso-aggregate particles

of latex are very small, the greater part of them being invisible under the ultramicroscope; the constituent parts of serum (*i.e.*, the dispersion medium in which the iso-aggregate particles are suspended), *viz.*, protein, water, resin, etc., are present not only in the surface film, but also inside the particles. These non-caoutchouc components play corresponding rôles in the changes the iso-aggregates undergo in their consistency and structure, during the drying-up and during other coagulation processes of latex. When latex is mixed with solutions of substances which are dispersators of protein, but without effect on caoutchouc, complete gelatinisation of latex into a coherent elastic material occurs. Elastic films of vultex are caused by the union of the caoutchouc contents of particles and not by the union of the non-caoutchouc layers. J. S. CARTER.

Effect of various types of carbon black on certain physical properties of rubber compounds. D. J. BEAVER and T. P. KELLER (Ind. Eng. Chem., 1928, 20, 817—819).—Acetylene black, thermatomic black, lampblack, and five varieties of carbon black are tested as to density, iodine adsorption, oil adsorption, acetone extract, oxidation by 0.02*M*-permanganate, and calorific value. The oxygen content of the various blacks, as indicated by differences in the inverse of the calorific value, causes a corresponding retardation in the rate of vulcanisation, decrease in the maximum physical properties attainable, and increase in the rate of ageing. It is therefore expected that in rubber mixtures thermatomic black and acetylene black with a low oxygen content will give better ageing results than lampblack or standard gas blacks with higher oxygen content. None of the other analytical features shows any correlation with the effect on ageing. D. F. TWISS.

Organic rubber colours. W. J. S. NAUNTON (Trans. Inst. Rubber Ind., 1928, 4, 68—84).—Rubber colours should be tested as to moisture content, fineness, freedom from water-soluble colour, shade, strength, light-fastness, and bleeding; they should also be tested under the exact conditions intended. Using a standard mixing of pale crêpe rubber 100 pts., zinc oxide 6 pts., lithopone 22 pts., catalpo 50 pts., magnesia 2 pts., sulphur 2 pts., tetraethylthiuram disulphide $\frac{3}{8}$ pt., and sufficient of various colours to impart a full shade, with vulcanisation for 10 min. at 40 lb. steam pressure, the colours appeared to have a bad influence on ageing at 70° but a good effect on normal ageing; in the additional presence of an anti-ager (nonox 1 pt.), even the heat-ageing was unaffected. Ageing had little influence on the actual colour of the mixed rubbers. Organic colours are classified into those (a) soluble in rubber, (b) slightly soluble in rubber, and (c) insoluble, and the characteristics of each group are given. The selection of colours for use with latex or in soft rubbers or ebonite, or for the production of transparent or "jazz" effects is indicated. D. F. TWISS.

Softeners and anti-softeners [for rubber]. E. C. ZIMMERMAN and L. V. COOPER (Ind. Eng. Chem., 1928, 20, 812—813).—Fluxes for rubber may be classified into lubricants and true softeners. For the evaluation of

a softener, a standard sample of raw rubber masticated under definite conditions is tested in a Williams plastometer (cf. B., 1924, 480) before and after the incorporation of 5% of the softening agent. Certain substances, *e.g.*, benzidine, *p*-aminophenol, and the naphthylamines, are anti-softeners, and cause a stiffening of unvulcanised stock. D. F. TWISS.

Classification of [organic] accelerators [of vulcanisation of rubber]. R. P. DINSMORE and W. W. VOGT (Trans. Inst. Rubber Ind., 1928, 4, 85–106).—The chemical nature of the commoner organic accelerators is indicated together with their practical features, such as their relative activity, tendency to produce scorching, best proportion of sulphur for vulcanisation, influence on tensile strength and extensibility of "unloaded" and black-compounded mixings, persistence during vulcanisation, antioxidant effect, influence on alteration in extensibility on storage, need for simultaneous presence of stearic acid, and effectiveness for vulcanisation in hot air. The decisions as to vulcanisation criteria were based on the hand-tear test (Dinsmore and Zimmerman, B., 1926, 289). In unloaded stocks, especially when vulcanised with sulphur only, but also in the presence of an accelerator, the discrepancy between the vulcanisation optimum as decided by hand-tear and tensile product is great, the former placing the optimum at a much shorter time; for loaded stocks the two methods show fair agreement.

D. F. TWISS.

Machine for testing rubber products used to absorb vibration. F. D. ABBOTT (Ind. Eng. Chem., 1928, 20, 853–857).—The ordinary physical characteristics, such as tensile strength, ultimate elongation, and permanent set after extension or compression, fail to give a satisfactory index of the resistance of vulcanised rubber to dynamic fatigue from flexure under compression. A machine is described for the testing of shock-insulators etc. which submits compressed samples to flexure at a rate of 800 cycles/min.; in these circumstances the permanent set approaches a maximum in 3 or 4 hrs., the temperature also rapidly attaining a maximum. The results place stocks in the same order as service and dynamometer tests.

D. F. TWISS.

Introfiers. DARRIN.—See III. **Stress-strain curves for rubber.** STRONG.—See VII.

PATENTS.

Electrodeposition of rubber or the like. W. A. WILLIAMS (B.P. 293,815, 8.4.27).—The process of B.P. 289,965 (B., 1928, 494) is extended to latex which has been previously vulcanised and to which chemicals or fillers may have been added. D. F. TWISS.

Preservation and treatment of latex. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 294,412, 12.11.27).—Soluble salts of sulphonic acids possessing soap-like qualities, *e.g.*, of aromatic or hydroaromatic sulphonic acids containing substituent alkyl, cycloalkyl, aralkyl, or aryl groups, or of sulphonic acids yielded by aliphatic tar oils, mineral oils, or fatty acids, are used for the preservation of latex. The presence of such a salt, *e.g.*, sodium butyl-naphthalenesulphonate,

alone or in conjunction with ammonia, is advantageous when latex is used for impregnating porous materials, *e.g.*, fabrics, cork, or paper. D. F. TWISS.

Treatment of rubber latex. L. MELLERSH-JACKSON. From GEN. RUBBER CO. (B.P. 294,002, 25.8.27).—Rubber latex, preferably containing a little ammonia, is treated with a small proportion of ammonium alginate solution. In 24 hrs. separation occurs with formation of a cream containing practically all the rubber in a concentrated but uncoagulated form; the lower layer contains the greater portion of the soluble non-rubber constituents. The cream can be converted into rubber by known methods, *e.g.*, spraying. D. F. TWISS.

Production of a reversible paste from rubber latex. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 294,804, 10.10.27).—Rubber latex is mixed rapidly with sufficient acid, *e.g.*, 7% hydrochloric acid, to give a p_H value below 2 and preferably below 1. After a period, *e.g.*, 24 hrs., the finely-divided deposit of rubber is removed and constitutes a reversible paste which can be stored unchanged in closed vessels. Other substances of importance for the further treatment of the rubber may be added either before or after the addition of acid. D. F. TWISS.

Galvanoplasty (B.P. 273,664).—See XI.

XV.—LEATHER; GLUE.

Determination of sulphuric acid in vegetable [tanned] leather. R. F. INNES (J. Soc. Leather Trades' Chem., 1928, 12, 256–272).—Free sulphuric acid in vegetable-tanned leather is determined by combining results with the Procter-Searle method (cf. B., 1927, 283) with those obtained by the following gravimetric and physical methods. The former consists in extracting 2 g. of the leather with 400 c.c. of 0.2*N*-sodium bicarbonate solution for 24 hrs. at laboratory temperature, and determining the total sulphates in the extract by means of barium chloride. In the physical method 1 g. of the leather is treated with 50 c.c. of water free from carbon dioxide for 24 hrs. with occasional shaking, and the p_H value of this aqueous extract is determined. A portion of the extract is diluted ten times, and the p_H value of the diluted extract is determined. If the difference between these two values is 0.5 or below, no strong mineral acid is present, but if it is 0.7 or above then it is concluded that strong mineral acid is present. Experiments on pelt and leather treated with sulphuric acid and afterwards extracted with water and sodium bicarbonate solution, respectively, showed that only the sodium bicarbonate removed the whole of the acid.

D. WOODROFFE.

Determination of insoluble matter [in tannin] by filtration in presence of kaolin. A. JAMET and A. J. GIRARD (J. Soc. Leather Trades' Chem., 1928, 12, 279–281).—The amount of insoluble matter as determined by the official method is shown to vary with the kaolin and also with its degree of fineness. The apparent insoluble matter increased when the kaolin used had been ground in an agate mortar. Other determinations with sieved kaolin gave variations of 2.45–4.1% in the values obtained with fine kaolin (300-mesh) and the residue from 230-mesh. D. WOODROFFE.

Tannin analysis. J. PAESSLER (Collegium, 1928, 352—361).—The hide powder is chromed, washed, and used for detanninisation without removal from the container—a small cylindrical glass vessel tapering slightly at its closed end and fitted with a stopper and tube at the mouth. The lower end of tube is attached to a small Berkefeld filter candle inside the container, the other end being attached to a filter pump. The vessel and powder are weighed together, and, after chroming and washing, the wash water is filtered off until the weight of wet chromed powder corresponds to a content of 20 g. of water. This method avoids handling the powder.

D. WOODROFFE.

Apparatus for determining the ethyl acetate value of tannins. C. RIESS (Collegium, 1928, 275).—A flask containing the tannin solution is in the form of a flat-bottomed tube. During the extraction condensed ethyl acetate drops into a funnel, the point of which leads into a cavity in the bottom of the tube, closed by a rough glass filter which will disperse the solvent into very fine droplets, thereby considerably increasing its surface and facilitating tannin extraction.

D. WOODROFFE.

Grading of commercial gelatin, and its use in the manufacture of ice cream. D. C. CARPENTER, A. C. DAHLBERG, and J. C. HENING (Ind. Eng. Chem., 1928, 20, 397—406).—Methods of grading commercial gelatin include measurement of mutarotation, gel strength, and viscosity, and also formol titrations. Experiments on purified, de-ashed, calfskin gelatin show that mutarotation is dependent to some extent on p_H . For bone and calfskin gelatins a p_H of 7.3 is satisfactory, but, owing to the isoelectric point of pigskin gelatin being close to this value, p_H 5 is selected in order to avoid the opacity of such solutions. For purified de-ashed calfskin gelatin, the gel strength at 0.7° and 10°, as measured by the Bloom gelometer (cf. Richardson, B., 1923, 413 A), shows a maximum value over the range, p_H 5—7. By lowering the hardening temperature from 10° to 0.7° the gel strength in this region is increased about 55%. For both calfskin and pigskin gelatins, the relation of gelatin concentration to gel strength at low temperatures is given by the equation: $s = KC^n$, where s is the Bloom strength, C the gelatin concentration (%), and K and n are constants. With longer time and lower temperature of hardening the gain in gel strength is greater with the lower-grade gelatins, especially for pigskin gelatin. Since increase in gel strength is also most marked in the less concentrated gels, the Bloom test should be made at 0.7° in gelatins to be used in ice cream. Addition of ammonium sulphate increases gel strength only slightly. Commercial pigskin gelatin gained materially in strength on adjustment of the p_H from the original acidity to 6.6, which is the p_H of milk and cream. The relation between Bloom strength and mutarotation for pigskin is linear, but for calfskin gelatins it is linear only at high gel strengths; hence mutarotation is not a specific property of the chemical species causing gelation. There is evidence also of a relationship between Bloom strength and viscosity, but the former is the more reliable index. The grading of commercial gelatins by means of formol titrations is unreliable, and suggested

changes in the method for use in ice-cream manufacture are discussed. Calfskin gelatin appears to exist in two forms: the sol form stable at 40°, in which the amino-acids are probably in chains, and the gel form stable at 0.5° which contains ketopiperazine linkings.

W. J. BOYD.

Pure food gelatin. Physical properties related to economy of manufacture. M. BRIEFER and J. H. COHEN (Ind. Eng. Chem., 1928, 20, 408—413).—A method has been devised for measuring the consistency of jellies which are too weak to permit the finger test or Bloom gelometer to be applied. A very small shot of definite weight is allowed to fall from a fixed height through the jelly, in which the fall is arrested at different levels according to the consistency of the jelly. The method of measuring turbidity is described, and the effect of p_H on jelly consistency and turbidity is studied, the four types of gelatin used being extracted from (a) acid-plumped pig skins, (b) limed splits, (c) limed sinews, and (d) limed pieces. The jelly consistency- p_H curves have each two maxima, but the depressions for limed-stock gelatins occur at about p_H 5, and for acid-plumped stock at about p_H 8. For pig-stock gelatin a 63% jelly tested by the Bloom gelometer shows a maximum at the p_H at which the jelly consistency shows a depression, a 0.76% jelly being used for the latter test. For gelatins of class (a) there is a turbidity maximum at p_H 8, whilst for the limed stock (classes b—d) the maximum occurs at p_H 4.9. The two types of gelatin cannot be mixed at any useful p_H value without producing a turbid solution. The significance of viscosity of the gelatin in relation to the sp. gr. of marshmallow cream has been studied. Experiments with gelatins, all of 200 g. Bloom but of different viscosities, reveal a steady rise in sp. gr. with decreasing viscosity. Gelatins of different jelly strengths but approximately equal in viscosity were then used to show the relation of jelly strength to sp. gr. Thus for a gelatin testing 140 g. Bloom it was necessary to increase the concentration much above the value based on jelly strength in order to give a marshmallow of the same sp. gr. obtained on that basis with a gelatin testing 200 g. Bloom.

W. J. BOYD.

Effect of previous history on the viscosity of gelatin solutions. C. E. DAVIS and H. M. SALISBURY (Ind. Eng. Chem., 1928, 20, 829—831).—A method is described for determining the viscosity of gelatin solutions. Owing to a gradual transition of the solutions to the gel form at 25°, reproducible results could not be obtained at this temperature, and the bulk of the measurements were therefore made at 40°. Viscosity- p_H curves are shown for a number of gelatins of which the sources of the raw material and the method of extraction are known. Gelatins of greatest jelly strength produce the highest viscosity- p_H curves. The maximum viscosity occurs in all cases at p_H 2.5—2.6. The short duration and low temperature of heating required to dissolve the gelatin has no effect on its viscosity.

F. R. ENNOS.

Dipping refractometer for determining the concentration of dilute glue liquors. A. C. HART (Ind. Eng. Chem., 1928, 20, 870—871).—The composition of

dilute glue liquors is usually inferred from their sp. gr., a method which does not give an accuracy of more than $\pm 0.5\%$. Greater accuracy is obtained by the use of the Zeiss dipping refractometer. A number of determinations of n for known solutions were made with this instrument, and the average value for the specific refractivity of glue was determined as 0.00183. Variations which were not correlated to the ash constant were found between different classes of glues. If the grade of glue is known determinations by this method should be accurate to $\pm 0.1\%$.

C. IRWIN.

"Zair" process. BROWN.—See VI.

PATENTS.

Treatment of skins containing calcified formations. J. PAISSEAU and R. A. GERMAIN (B.P., 272,199, 27.5.27. Fr., 3.6.26).—Skins, such as crocodile skin, with calcareous tubercles, are macerated in an acidic liquid adopted to convert the calcareous substances gradually into soluble diffusible salts without harm to the derm; fixing or tanning agents are also included in the bath in order to assist the protection of the derm. The skin is then washed and tanned. Glycerin or deliquescent salts may be added to the acid bath or the tanning bath to prevent contraction of the decalcified nodules during drying.

D. F. TWISS.

Preparation of a cold glue powder. G. HÖNSCH (B.P. 274,490, 14.7.27. Ger., 16.7.26).—Finely-powdered animal glue is mixed with a colloid-liquefying substance of a neutral or weakly acid character, e.g., solid organic acids or their salts, calcium nitrate, etc., and with a drying or dressing agent, e.g., magnesium carbonate, alum, or quartz powder.

F. R. ENNOS.

Arylaminesulphonic acid derivatives (B.P. 293,781).—See III.

XVI.—AGRICULTURE.

Colloidal behaviour of soils and soil fertility.

IV. Anion effect on precipitation reactions and degree of dispersion of aluminium and iron hydroxides. J. S. JOFFE and H. C. McLEAN (Soil Sci., 1928, 26, 47—59; cf. B., 1927, 308).—The solubility of aluminium and iron and the formation of the different states of aggregation are controlled not only by the hydrogen-ion concentration, but primarily by the anions present. Experiments on sol and gel formation in solutions of iron and aluminium salts showed that gel formation with salts of aluminium occurred at the following p_H values for various anions: sulphate 4.7—4.8, chloride 5.4, nitrate 5.8—6.0. With iron, corresponding values are: 3.2—3.8, 5.4, 5.4. In mixtures of anions the properties of bivalent or trivalent anions control the states of aggregation of the aluminium and iron colloids. This almost precludes the existence of free iron in molecular state in normal soils.

H. J. G. HINES.

Determination of exchangeable calcium in carbonate-free soils. R. WILLIAMS (J. Agric. Sci., 1928, 18, 439—445).—Exchangeable calcium can be determined satisfactorily by using 0.5*N*-acetic acid as the leaching agent.

H. J. G. HINES.

Potash manures and their influence on soils. A. STROBEL and W. SCHROPP (Illustr. Landw. Ztg., 1927, 47, 642; Bied. Zentr., 1928, 57, 255—257).—Poppies, potatoes, beet, and cabbages benefited considerably from potash manuring in a 12-year experiment, but straw crops were not greatly affected. No harmful effect of potash salts on the reaction, physical properties, or the microflora of the soil could be observed.

H. J. G. HINES.

Factors affecting the iron and manganese content of plants, with special reference to herbage causing "pining" and "bush-sickness." W. GODDEN and R. E. R. GRIMMETT (J. Agric. Sci., 1928, 18, 363—368).—Preliminary pot experiments with oats and mustard are reported using New Zealand soils on which the disease known as "pining" was prevalent. These soils were compared with a local soil (Aberdeen), and with sand with various manurial treatments. It is indicated that lack of drainage is an important factor in increasing the manganese content of the crop.

H. J. G. HINES.

A "deficiency disease": lack of available manganese in a lime-induced chlorosis [in crops]. B. E. GILBERT and F. T. McLEAN (Soil Sci., 1928, 26, 27—31).—"Yellowing-off" of maize, onions, and mangolds was prevented by treatment with manganese salts, preferably in a liquid form.

H. J. G. HINES.

Assimilation of biogenic elements by the roots of crops. J. STOKLASA (Vestn. Českoslov. Akad. Zemedelske, 1927, 3, 242—255; Bied. Zentr., 1928, 57, 259—262).—Difficultly soluble substances in the soil are brought into solution by acid excretions from plant roots, organic acids and carbonic acid both being excreted. The carbon dioxide produced by microbiological action also plays an important part in rendering insoluble materials soluble.

H. J. G. HINES.

Increase of iodine content of plants. E. HILTNER (Forts. Landw., 1928, 3, 1; Bied. Zentr., 1928, 57, 258—259).—In an experiment on meadow land using potassium iodide as a source of iodine, the values obtained for the iodine content of the dry matter were: untreated, 0.146; manured with potassium iodide, 0.797; sprayed with potassium iodide solution, 2.351 mg./kg.

H. J. G. HINES.

Composition and metabolism of the tobacco leaf at different stages of growth of the plant. A. SMIRNOV and co-workers (Ber. Centr. Inst. Tabakforsch. Krasnodar, 1926, 29; Bied. Zentr., 1928, 57, 264—265).—A complete account of the distribution of organic substances in the leaves is given for four stages in the life of the plant, viz., at planting, at the stage of 8—10 leaves, in full bloom, and at the technically ripe state of the leaf.

H. J. G. HINES.

Growth of grapes. IV. Initial changes in acidity. V. Relationship between sugar and soluble solids in the juice. VI. Acid: sugar ratio. P. R. v.D. R. COPEMAN (Trans. Roy. Soc. S. Africa, 1928, 16, ii, 103—106, 107—113, 115—120; cf. A., 1927, 908).—The above relationships have been expressed in definite mathematical forms.

H. J. G. HINES.

Compound and complete fertilisers. A. MITRASCU (Z. angew. Chem., 1928, 41, 902—916).—Compound fertilisers may be classified as (1) superphosphates with added nitrogen or potash, (2) organic residues used mainly in America, (3) fertilisers developed by the synthetic nitrogen industry. The last-named include "Kaliamonsalpeter" (a mixture of potassium nitrate and ammonium chloride in equimolecular proportions), "Leunaphos" or diammonium phosphate, now chiefly prepared from phosphoric acid made in the electric furnace, and "Nitrophoska," prepared by adding to fused ammonium nitrate diammonium phosphate and potassium chloride or sulphate. Five different mixtures are prepared, according to the soil and requirements of the crops. Another group contains the nitrogen as urea. The microscopical examination of these mixtures is described, and solubility diagrams and vapour-pressure curves of the saturated solutions of the salts concerned are given. In the study of caking not only the rate of water absorption from the air, but the amount of water required to cause caking must be considered. The total "water capacity" is determined by tests in air of known moisture content. An apparatus is illustrated which determines the "capability of distribution" of a fertiliser, i.e., the quantity spread over a given area. The caking pressure may also be determined. The proportioning of nitrogen, phosphorus, and potash in the plant substance, in animal manure, and in Nitrophoska is compared. It is claimed for Nitrophoska that all its ingredients are readily assimilated, that the mixture is perfectly uniform, and that it can be stored and sown in any climate and at any time of the year. C. IRWIN.

Field experiments on the availability of nitrogenous fertilisers, 1923—1927. J. G. LIPMAN, A. W. BLAIR, and A. L. PRINCE (Soil Sci., 1928, 26, 1—25).—In discussing the results for the last five years of this twenty-year old experiment emphasis is laid on the following points. Great difficulty is experienced in maintaining the nitrogen supply of the soil where no legumes are grown. Under field conditions the recovery of applied nitrogen is very low (about 30%), and great losses of nitrogen occur. Liming is imperative where physiologically acid fertilisers are used. Mineral nitrogenous manures have proved superior to the organic forms. H. J. G. HINES.

Nitrogen fixation by soil micro-organisms. P. G. KRISHNA (J. Agric. Sci., 1928, 18, 432—438).—*Azotobacter* and *B. amylobacter* groups are the important agents in the fixation of nitrogen in soil, their optimum ranges being p_H 7.0—8.4 and p_H 6.0—7.0, respectively. Fungi are responsible for the fixation of small amounts of nitrogen. H. J. G. HINES.

Bordeaux mixture in combination with arsenical sprays. W. GOODWIN and H. MARTIN (J. Agric. Sci., 1928, 18, 460—477).—The interaction of Bordeaux mixture with lead arsenate and with calcium arsenate was studied by examining the effects produced (a) by adding hydrated lime to the arsenical compounds, (b) by adding copper sulphate to the arsenical compounds and hydrated lime. Under conditions involving the

long-continued action, in the presence of water, of calcium hydroxide on lead arsenate, the latter is decomposed with the formation of basic calcium arsenates which in the presence of water and carbon dioxide are converted into the relatively soluble calcium monohydrogen arsenate. If, however, the conditions are such that the carbonation of the excess of calcium hydroxide occurs with sufficient rapidity, the amount of soluble arsenate compounds formed is not sufficient to make the liability to arsenical injury greater. It is concluded that when the conditions are such that the addition of hydrated lime brings about a reduction of arsenical injury, the use of an "equal lime" Bordeaux mixture containing an equivalent amount of calcium hydroxide will prove far more effective.

H. J. G. HINES.

Petroleum oil as carrier for insecticides and as plant stimulant. E. R. DE ONG (Ind. Eng. Chem., 1928, 20, 826—827).—Attention is drawn to the increased penetrating, spreading, and wetting powers of nicotine and other insecticides when used in combination with emulsions of fractions of petroleum oils which are not injurious to foliage. Some physiological effects of oil sprays on fruit trees are also discussed.

C. T. GIMMINGHAM.

Analysis of insecticides. Liquid insecticides miscible with water: alcohol, mercury and formaldehyde, picric acid, nicotine, and garlic essence. M. FRANÇOIS and (Mlle.) L. SEGUIN (J. Pharm. Chim., 1928, [viii], 8, 105—112; cf. B. 1928, 332).—Methods for the detection and determination of nicotine, picric acid, formaldehyde, mercuric chloride, and ethyl alcohol in liquid insecticides are given. E. H. SHARPLES.

Miscible carbon disulphide. FLEMING and WAGNER.—See III.

PATENTS.

Treatment of [manufacture of fertilisers from] material containing magnesium [and potassium chlorides]. F. G. LILJENROTH (B.P. 283,558, 26.4.27. Swed., 15.1.27).—A solution of a mixture of potassium and magnesium sulphates, prepared by treating the material, e.g., kainite, with sulphuric acid, is treated with ammonia and carbon dioxide, and, after removal of the precipitated magnesium carbonate, the liquor, which contains potassium and ammonium sulphates, is used for precipitating calcium from the liquor obtained by leaching phosphatic rocks with nitric acid, yielding a fertiliser containing nitrogen, potassium, and phosphoric acid, but practically free from sulphuric acid, chlorides, and magnesium. L. A. COLES.

Manufacture of ammonia phosphate fertiliser. AMER. CYANAMID CO. (B.P. 284,322, 16.1.28. U.S., 28.1.27).—Dry monoammonium phosphate is treated with gaseous ammonia in a closed container at temperatures (preferably 60—150°) and pressures above atmospheric. The granules in this way become coated with triammonium phosphate. On storage, diammonium phosphate is formed throughout the mass, and any excess ammonia is removed by evacuation.

H. J. G. HINES.

Fertilisers (B.P. 276,297).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Recovery of potassium salts and other alkaline substances in the sugar industry. T. G. Y ARNAL (Chim. et Ind., 1928, 20, 27—29).—Molasses is treated with a solution of calcium ferrocyanide which reacts with the potassium salts present to form the slightly soluble ferrocyanide of calcium and potassium. After filtration a further yield of sugar crystals may be obtained from the syrup. For the recovery of the reagents and salts, the filter cake is mixed with a solution of ferric chloride or sulphate, preferably the latter, and the Prussian blue and calcium sulphate formed are separated from the soluble potassium sulphate and treated with slaked lime. The soluble calcium ferrocyanide is recovered, and to the ferric hydroxide and calcium sulphate is added the theoretical quantity of sulphuric acid to convert the former into ferric sulphate, which is separated by decantation. F. R. ENNOS.

The Laurent saccharimeter. W. ANDERSON (J. Mun. Coll. Tech. Manchester, 1928, 13, 158—160).—The instrument may be used to determine the rotation of light of any colour, but its sensitiveness will depend on the phase difference produced by its crystal for that colour. The sensitiveness of the instrument is reduced in the ratio $(1 - \cos \phi)/2$ when the crystal produces a phase difference ϕ , compared with the ideal phase difference π . F. G. TRYHORN.

New soluble starch and an improved polarimetric Lintner method. H. C. GORE (Ind. Eng. Chem., 1928, 20, 865—866).—Starch is treated with 13% hydrochloric acid at room temperature for 6 days, washed free from acid, and dried in a current of warm air. By cooling the hot 6% solution, keeping it, and filtering, a clear permanent solution of about 5% concentration is obtained, the use of which greatly increases the accuracy of Lintner's method for the determination of diastatic activity, since a polarisation fall of at least 11.3° V. is permissible as against 3° V. with the 2% solution of soluble starch in the earlier method (cf. B., 1924, 648). F. R. ENNOS.

Locust-kernel gum and oil. A. L. WILLIAMS (Analyst, 1928, 53, 411—415).—Commercial "locust-kernel gum" or "gum tragon" is a nearly white powder made from the heated endosperms of *Ceratonia siliqua*, which are stirred in about twenty times their weight of boiling water, filtered, and dried. "Tragasol" is a tough jelly containing about 4% of solids. The composition of the gum calculated on the ash-free substance present agreed with the formula $C_6H_{10}O_5$, i.e., it is a carbohydrate composed of the anhydrides of the hexoses mannose and galactose, and probably belonging to the hemicelluloses. The gum is precipitated by a dilute solution of tannin. The soft gelatinous mass formed shrinks on keeping and finally separates as a white or buff-coloured clot and a clear supernatant liquid, but disperses on warming and reappears on cooling. Precipitation occurs in acid but not in alkaline solutions, but not in the presence of acetic acid. Industrial uses of the gum are indicated. It may be detected by its reaction with tannin, borax, and Fehling's solutions. The extracted dark-green oil from a sample of old and fresh locust kernels had: d_{20}^{25} 0.950, 0.951; butyro-

refractometer reading at 40° 65.0, 65.0; iodine value (Wijs) 98.5, 99.1; saponif. value 205.5, 198.0; unsaponifiable matter, —, 2.86%; insoluble fatty acids 86.5, 87.4%; Reichert-Meissl value, —, 1.8; Polenske value, —, 0.8; titre 25.4—25.7°, 25.2—25.6°; iodine value of insoluble fatty acids 101.7, 100.5. The ground kernels contained water 12.08; oil 1.80; ash 2.80; proteins 15.12; and fibre 6.10%.

D. G. HEWER.

Sugar-beet pulp. WOODMAN and CALTON.—See XIX.

PATENTS.

Production of sugar from dried beet. SUGAR BEET & CROP DRIERS, LTD., W. K. MELROSE, and J. C. STEAD (B.P. 293,946, 24.5.27).—The raw syrup obtained by extracting dried beets with water at 50—70° is treated with lime (0.08—0.16% by wt. of the beets) and clarified by a centrifuge. After increasing the sugar content to 60—65% by addition of low-grade or second-product sugar from a later stage, the liquor is decolorised by addition of 2—4% of activated carbon and crystallised. F. R. ENNOS.

Purification of sugar solutions. F. W. MEYER (U.S.P. 1,678,571, 24.7.28. Appl., 10.5.27. Ger., 7.7.26).—The solution is treated, at 90°, with lime until free from colour, neutralised, filtered, and the surface tension increased by addition of carbon.

B. FULLMAN.

Manufacture of dextrose. C. EBERT, W. B. NEWKIRK, and M. MOSKOWITZ, Assrs. to INTERNAT. PATENTS DEVELOPMENT Co. (U.S.P. 1,673,187, 12.6.28. Appl., 1.4.27).—"Hydrol," the mother-liquor obtained in the ordinary process for the manufacture of solid dextrose, is neutralised until the impurities which cause polymerisation of the dextrose are coagulated and precipitated. The filtered liquid is then hydrolysed with sufficient acid in a vessel the inner surfaces of which are catalytically inert, neutralised, filtered mechanically or through carbon, and evaporated to crystallisation.

F. R. ENNOS.

Starch size (U.S.P. 1,677,614—5).—See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

Origin of yeast. J. GRÜSS (Woch. Brau., 1928, 45, 341—344, 353—357).—A description of certain fossil forms from the Devonian of Spitzbergen, Neunkirchen, etc., which appear to be the remains of yeast-like organisms. F. E. DAY.

Volumetric determination of carbon dioxide in beer. H. LUNDIN and J. ELLBURG (Woch. Brau., 1928, 45, 339—341, 349—352).—By means of a cork-borer of special construction, the beer is transferred to a measuring flask without loss of carbon dioxide. The flask already contains an excess of carbonate-free caustic soda, and the alkaline beer is made up to volume. The carbon dioxide is determined on 1 c.c. of the liquid by addition of sulphuric acid in a Van Slyke blood-gas apparatus. It is claimed that a determination can be completed in 15—20 min., the accuracy being about 1% of the amount present. F. E. DAY.

Photometric determination of colour of malt worts. F. MĚŠTAN (Woch. Brau., 1928, 45, 373—377).—An improved form of Zeiss' "step photometer" is

described in which Ostwald's grey filters are replaced by Aubert's adjustable apertures, one in front of each objective. These are each operated by a drum graduated in degrees, which scale gives a finer division for low than for high intensities. By the use of suitable colour filters the absorption (colour) of malt worts can be very accurately determined, and is stated to vary by about 4% for a colour difference corresponding to 0.01 c.c. of 0.1*N*-iodine for colours equivalent to 0.15–0.17 c.c. of 0.1*N*-iodine per 100 c.c. The colour of beer may be similarly determined. The slightest opalescence necessitates the use of a special nephelometric addition to the apparatus.

F. E. DAY.

Objective determination of colour of malt worts. F. DUCHÁZEK (Woch. Brau., 1928, 45, 359–362, 369–373).—For the determination, Sandera's "universal objective photometer" (B., 1928, 344) has been employed. Using approximately monochromatic screens, it is found that the absorption curves for different wavelengths are similar in the cases of iodine, wort, beer, and a standard solution containing potassium dichromate and cobalt sulphate. By using 100 c.c. of wort, colour differences of 0.015 c.c. of 0.1*N*-iodine can be certainly distinguished at the most important range, about 0.15 c.c. of 0.1*N*-iodine, the millivoltmeter showing a difference of 4 scale divisions for blue light, or 2–3 divisions when white light is used. The possibility of increasing this sensitivity by the use of a deeper layer of liquid or by increased concentration of laboratory worts is discussed. In the latter connexion the colour is shown to be directly proportional to the concentration.

F. E. DAY.

Fermentation products. III. Effect of vessel on the constituents of "saké." T. HIGASI (Bull. Inst. Phys. Chem. Res., Japan, 1928, 7, 763–775).—The effect of aqueous and alcoholic extracts of "sugi" wood (*Cryptomeria Japonica*) on Japanese "saké" has been studied. The distillate of an alcoholic extract of sugi shavings contains acetaldehyde, the increase in the aldehyde content of saké during storage being due to the catalytic oxidation of alcohol by the sugi wood. The extract and especially the distillate also contain a sesquiterpene "cryptene" (Keimatsu, J. Pharm. Soc. Japan, 1905, No. 277, 189) and a substance which gives similar colour reactions to those obtained with the oxidation product of protocatechuic aldehyde or vanillin with hydrogen peroxide, and is probably a hydroxy- or methoxy-quinol.

J. W. BAKER.

Detection of isopropyl alcohol [in wine]. D. HENVILLE (Analyst, 1928, 53, 416–418).—In order to test wine for added methylated spirit a 10% alcoholic solution of the sample after distillation is made. The presence of isopropyl alcohol is detected by the presence of acetone after oxidation. To a mixture of 10 c.c. of water and 5 c.c. of sulphuric acid when cool, 10 c.c. of the alcoholic solution are added. After addition of 3 g. of potassium dichromate, the mixture is shaken, left for 5 min., and the contents are distilled; to the first 2 c.c. of distillate are added 2 c.c. of water and 1 c.c. of acetic acid (B.P.) followed by 2–3 drops of a strong fresh solution of sodium nitroprusside. Ammonia solution is added in excess to a total volume of about

8 c.c. and a violet coloration is produced either at once or on keeping, according to the amount of acetone present. Industrial methylated spirits contain about 0.5% of acetone. The test will detect 0.1% of isopropyl alcohol in the prepared solution.

D. G. HEWER.

Improved Lintner test. GORE.—See XVII. **Treatment of distillery slop.** NEAVE and BUSWELL.—See XXIII.

PATENTS.

Production of yeast. S. SAK (B.P. 294,123 and 294,131—4, 14.1.27).—(A) During the fermentation a predetermined or constant weight of yeast per unit volume of wort is established for a short or long time by a controlled return to the fermenting wort of part or all of the yeast centrifuged from the yeast-containing wort withdrawn during the fermentation. (B) The yeast which is added to the fermenting wort may originate from a source other than the fermenting wort. (C) The desired weight of yeast per unit volume of fermenting wort may be maintained by the addition during fermentation to the fermenting wort of fresh wort and the withdrawal of yeast-containing wort. (D) During the fermentation the yeast-containing wort is withdrawn and centrifuged, and the centrifuged wort, after complete or partial removal of its content of alcohol by distillation, in some cases under reduced pressure, is returned continuously to the fermenting wort. (E) Part or all of the centrifuged yeast from a fermenting wort is transferred to another fermentation vat containing fermenting wort, the concentration of which with regard to nutrients and salts is higher than the concentration of the same substances in the initial vat. After the yeast has displayed a more or less intensive activity of growth in the second vat, it is returned in the form of centrifuged yeast to the initial vat.

C. RANKEN.

Production of glycerol by fermentation. J. W. LAWRIE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,678,150, 24.7.28. Appl., 27.7.22).—A mixture of substantially the same composition as the incinerated ash of a fermented molasses mash is added to a molasses mash during its fermentation by yeast.

C. RANKEN.

Making a size or dressing. Prepared starch material. A. BODIN and J. EFFRONT (U.S.P. 1,677,614—5, 17.7.28. Appl., 24.2.25).—(A) Starchy material is subjected to the action of a suitable starch-liquefying bacterial enzyme, and, when the required amount of starch has been converted, further enzyme action is arrested. (B) A dry starch product suitable for making size is obtained by compounding a preparation of enzymes from micro-organisms capable of liquefying starch with a neutral starch and adjusting the relative moisture content of the enzyme preparation and the starch so that the final product contains a normal (air-dry) amount of moisture.

D. J. NORMAN.

Beverages (U.S.P. 1,673,273–5).—See XIX.

XIX.—FOODS.

Effect of alkali solutions on bacteria found in unwashed milk bottles. C. S. MUDGE and B. M. LAWLER (Ind. Eng. Chem., 1928, 20, 378–380).—To determine the influence of concentration, tempera-

ture, and time of contact on the germicidal action of the alkali used in milk bottle washing machines, a wash water was prepared by rinsing 40 dirty bottles each with 10—15 c.c. of sterile distilled water, and uniting the rinsings. Plate cultures were prepared from 1 c.c. portions of this, which had been subjected to the required conditions. The p_H of the alkali solution is a determining factor. A 0.5% solution of sodium hydroxide is almost as effective as a 2% solution of a commercial cleanser consisting of a mixture of sodium carbonate and hydroxide.

W. J. BOYD.

Viscosity and the ice cream mix. G. D. TURNBOW and K. W. NIELSON (Ind. Eng. Chem., 1928, 20, 376—378).—True viscosity is derived from the components in solution in the mix, but during normal ageing (24—48 hrs. at 1.1—4.4°) there develops an "apparent viscosity" due to the formation of a gel structure and believed to be necessary for proper yield and smoothness of texture. As the ageing is continued, incorporation of air into the mix during freezing becomes difficult. Although the apparent viscosity increases during ageing, it is reduced again to the viscosity of the fresh mix from the cooler by sufficient agitation. The benefits obtained by ageing are due not to the effect of increased viscosity, but rather to some change in the proteins in the mix. By repeated homogenisation the apparent viscosity is decreased, and although it still increases during ageing the relationship is maintained. Surface tension is not affected by ageing, but tends to increase slightly with successive homogenisation. Repeated homogenisation has no material influence on the total time for freezing and whipping nor on the texture of the final product.

W. J. BOYD.

Determining a satisfactory standard for beriberi-preventing rices. E. B. VEDDER and R. T. FELICIANO (Philippine J. Sci., 1928, 35, 351—389).—The relation between the beriberi factor (number of birds developing polyneuritis, expressed as a percentage of the total number used in the experiment, divided by average number of days before appearance of first symptoms) and percentage of pericarp remaining on the grain of polished rice, and percentages of moisture, fat, phosphorus, and ash is investigated with 200 different samples of rice, variously polished, fed with water to pigeons as sole food. The best and most rapid criterion of vitamin content is the percentage of remaining pericarp, estimated visually after staining the grains with an iodine solution: no rice with above 50% of pericarp remaining caused beriberi. As a chemical index nitrogen content is valueless, and ash content poor; a phosphorus minimum expressed as 0.62% of the pentoxide or a fat minimum of 1.28% is better. A minimum of 1.77% of phosphorus pentoxide plus fat (of which at least 0.4 is phosphorus pentoxide) is suggested as a standard. Washing the rice with cold water causes a fall of about 0.25 in the phosphorus percentage, and presumably also removes the water-soluble vitamin. Storage, even in a damp place, does not destroy the beriberi-preventing property, if insects are excluded (e.g., by use of chloroform or carbon tetrachloride), but in samples in which insects are allowed to live the above chemical indices fell, and polyneuritis of pigeons occurred with each sample.

E. W. WIGNALL.

Chemical examination of army biscuit. J. STRAUB and J. P. PEPER (Pharm. Weekblad, 1928, 65, 765—776).—The manufacture of the emergency ration biscuit for the Dutch army is discussed in the light of the specification laid down, and of analyses of the materials used and of the products.

S. I. LEVY.

Coffee parchment as adulterant of bran and sharps. J. EVANS and T. E. WALLIS (Analyst, 1928, 53, 432—434).—Coffee parchment—the thin and tough endocarp of the coffee fruit—in transverse section shows three or four layers of lignified sclerenchyma, each layer consisting of groups of cells with their long axes parallel to each other. When found in bran the particles of parchment are fairly large, of a pale buff colour, and appear homogeneous and semi-opaque; they are free from starchy matter, show no definite lines, ridges, or striations, and are stiff, hard, and usually slightly curved. In sharps the parchment is more finely comminuted, and if present in such a material in small quantities a crude fibre should be prepared, and the amount present calculated, since coffee parchment yields about 60% and bran and sharps about 10%.

D. G. HEWER.

Antirachitic properties of cod-liver meals. R. M. BETHKE, G. ZINZALIAN, D. C. KENNARD, and H. L. SASSAMAN (J. Agric. Res., 1928, 36, 747—753).—The antirachitic value of cod-liver meals (dried residues after extraction of the oil) is below that of the fresh oil. Experiments with rats and chicken show that the residual antirachitic properties vary considerably, but are not proportional to the oil remaining.

A. G. POLLARD.

Action of hydrocyanic acid on fruit and vegetables and its determination. K. AMBERGER (Pharm. Zentr., 1928, 69, 481—483).—Apples, pears, celery, and potatoes, after being exposed to the action of hydrocyanic acid, become soft, wrinkled, and pasty, and are found to contain 7—16 mg. of the acid per 100 g. of material. The distillation test is very unsatisfactory for the determination, probably because of the formation of cyanohydrins which hydrolyse to carboxylic acids. A colorimetric method, based on the formation of Prussian blue by extraction with cold water, addition of ferrous and ferric salts, and acidification, is described, the blank solutions being prepared from similar fruit or vegetable not subjected to the disinfection treatment.

S. I. LEVY.

Jelly-strength measurements of fruit jellies by the Bloom gelometer. C. R. FELLERS and F. P. GRIFFITHS (Ind. Eng. Chem., 1928, 20, 857—859).—The Bloom gelometer with a few minor modifications can be satisfactorily used for the determination of the jelly strength of fruit jellies, and for the standardisation of manufactured products and the grading of commercial pectins. The maximum increase in jelly strength occurs very soon after making the jelly, but a gradual change takes place over a period of several days. Jellies for testing should be sealed with paraffin, stored for at least 24 hrs., and the determination made at constant temperature, preferably 20—23°.

F. R. ENNOS.

Composition and nutritive value of sugar-beet pulp. H. E. WOODMAN and W. E. CALTON (J. Agric. Sci., 1928, 18, 544—568).—Sugar-beet pulp is shown to

be highly digestible in the ruminant organism. Drying does not depress digestibility, and the dried pulp can be regarded as a carbohydrate concentrate, 1 lb. of which is capable of replacing 0.8 lb. of maize or 0.9 lb. of barley in the productive part of the rations of ruminants. The digestion coefficient of the protein is relatively low, but that of the fibrous constituents and nitrogen-free extractives is very high. Almost four fifths of the total dry matter of the pulp is digested not by enzymic processes but by the agency of bacteria.

H. J. G. HINES.

Determination of iodine in mineral mixtures [for feeding stuffs]. M. D. KNAPHEIDE and A. R. LAMB (J. Amer. Chem. Soc., 1928, 50, 2121—2125).—The sample of animal food (10 g.) is fused with sodium hydroxide (20 g.) and potassium nitrate (10 g.), the solidified mass is dissolved in water (600 c.c.) containing syrupy phosphoric acid (10 c.c.), 10 c.c. of 20% sodium hydrogen sulphite solution are added, and the whole is heated at 100° and cooled. The solution is then neutralised with 85% phosphoric acid, 1.5 c.c. of which is added in excess, and after addition of a small piece of anthracite coal the mixture is evaporated to 400—500 c.c. Excess of bromine water is added, and the whole boiled until colourless. A few salicylic acid crystals are added, and after the addition of 5 c.c. of reduced phosphoric acid (Kendall, B., 1920, 707 A) and 0.5—1.0 g. of pure potassium iodide to the cooled solution it is titrated in the usual way with 0.005*N*-sodium thio-sulphate, standardised according to Kendall's method.

S. K. TWEEDY.

Detection and determination of starch in margarine. A. SCHMIDT (Milch. Zentr., 1928, 57, 213—214, and Chem.-Ztg., 1928, 52, 671).—The presence of starch may be detected by its reaction with iodine under the microscope or in the fat-free layer formed on melting the fat below 60°. For its determination the margarine is melted (below 60°), the fat removed, and starch in the residue, after digestion with acid, is determined polarimetrically. The Soltsien reaction (fuming stannous chloride) for sesame oil is preferred to the Baudouin test.

E. LEWKOWITSCH.

Identification of prohibited coal-tar colours in foodstuffs. A. R. JAMIESON and C. M. KEYWORTH (Analyst, 1928, 53, 418—423).—About 1.5 c.c. of the extract of colouring matter is placed in each of five test-tubes. The first is used to determine whether the dye is sulphonated or not. To the second 1 drop of berberine sulphate is added, a precipitate indicating either picric acid or Martius Yellow, the latter to be confirmed by the gold chloride test. If there is no precipitate or change of colour the contents of the tube are then tested for Victoria Yellow by the reduction test (2 drops of hydrochloric acid and 1 drop of Wijs' solution are added, the mixture is boiled, and granulated zinc added; after 12—48 hrs. a delicate pink forms in the presence of the dye). To the third tube a drop of phosphotungstic acid is added. A precipitate with discoloration may indicate Aurantia, which may be confirmed by the reaction with silicotungstic acid. The contents of the fourth tube are tested for Aurin, and the fifth is used for confirmatory tests. Precipitates are examined microscopically after being kept overnight,

and the results may be compared with the characteristic photomicrographs reproduced.

D. G. HEWER.

Gelatin for ice-cream. CARPENTER and others, also BRIEFER and COMEN.—See XV. **Ginger.** CLEVENGER.—See XX.

PATENTS.

Re-making cheese. S. K. ROBINSON, Assr. to KRAFT-PHENIX CHEESE Co. (U.S.P. 1,678,167, 24.7.28. Appl., 21.6.23).—The comminuted cheese is heated and mixed with a small amount of sodium potassium tartrate.

C. RANKEN.

Beverage extracts. Alcohol-reduced cereal beverages rich in proteins. L. WALLERSTEIN, Assr. to WALLERSTEIN Co., INC. (U.S.P. 1,673,273—5, 12.6.28. Appl., 13.8.20. Renewed [c] 4.4.28).—A mixture of malt with excess of wheat is mashed under such conditions of temperature and time that a high percentage of dextrin and a low percentage of fermentable sugars are produced. It is then (A) proteolysed at 50° by addition of an enzyme, filtered, and the filtrate evaporated to a thick syrup extract; (B) proteolysed, boiled with hops, and finished to form a beverage; (C) mixed with material rich in soluble non-coagulable proteins and converted into a beverage.

F. R. ENNOS.

Production of protein products. F. C. ATWOOD, Assr. to AMER. PROTEIN CORP. (U.S.P. 1,673,964, 19.6.28. Appl., 5.12.22).—After separation of all the red corpuscular matter from a quantity of animal blood, the solution is heated to 38° to ferment the blood sugar, separated from coagulated fibrinogen, and dried. By adding to the protein solution sufficient fibrinous material to form a froth when whipped, a product having the characteristics of egg-white is produced.

F. R. ENNOS.

Vitamin food products. V. F. FEENY. From VITAMIN FOOD Co. INC. (B.P. 293,735, 11.4.27).—Food-stuffs containing vitamin are protected from deleterious aerial oxidation by thorough admixture with a film-forming edible gum (e.g., karaya gum).

E. LEWKOWITSCH.

Purification of phosphatides. H. BOLLMANN, Assr. to M. F. FOSTER (U.S.P. 1,673,615, 12.6.28. Appl., 13.7.25. Ger., 13.6.25).—An emulsion of lecithin, phosphatides insoluble in alcohol, oil, bitter substances, and other impurities is diluted with 1 pt. of benzene and 9 pts. of alcohol, heated, and maintained at b.p. for some time. After settling, the clear solution therefrom is cooled to about 20° and the phosphatides insoluble in alcohol are removed. The remaining solution is evaporated in a vacuum to obtain the lecithin, which may be freed from oil and impurities by washing with acetone.

F. R. ENNOS.

Extraction of nutritive matter from fibrous vegetable materials. V. P. H. I. W. D'OBRY (B.P. 293,779, 11.4.27).—Grass, straw, etc. is subjected to the action of steam for 2—4 hrs. at 155—159° under 5—6 atm. pressure. After disintegration of the steamed mass it is mechanically worked into a form suitable for cattle food, or the liquid containing all the nutritive materials is separated by pressure.

F. R. ENNOS.

Treating fish, animal, or vegetable products. M. AVANZI (B.P. 269,923, 22.4.27).—The fish etc. is centrifuged to remove blood or juices, passed through steam cooking ovens and then through hot-air drying ovens, filled into boxes, impregnated with oil *in vacuo*, and sterilised in tubular autoclaves. B. FULLMAN.

Manufacture of chocolate. A. NYROP (B.P. 294,790, 14.9.27).—The usual materials are first liquefied. Thus the sugar is dissolved in the milk and the ground beans are swelled, or the roasted and peeled beans are swelled by being heated in water or milk and comminuted. The liquefied raw materials are atomised in hot air or other gas simultaneously with melted cocoa-mass by the same or different atomisers. The fine powder of chocolate coated with fat is subsequently moulded. The beans, in aqueous suspension, may be fermented or treated with alkali before the drying process to modify the flavour. B. FULLMAN.

Chewing gum. B. ARKELL (B.P. 294,299, 2.5.27).—1–3% of magnesium oxide or hydroxide, milk of magnesia, etc. is incorporated in chewing gum to neutralise acids in the mouth. B. FULLMAN.

[Steam-heated retorts for] preservation of food. ANCHOR CAP AND CLOSURE CORP., Assees. of G. W. MULLEN (B.P. 271,052, 11.4.27. U.S., 12.5.26).

Drying apparatus applicable to preparation of fish flour and the like. E. L. RICHARDSON and W. WEST (B.P. 294,914, 29.4.27).

[Spraying device for] treatment of air for preservation of foods etc. W. S. LEECH and A. E. SHERMAN (B.P. 294,586, 27.1.27).

Fats rich in vitamins (B.P. 293,777).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Effect of sunlight on citric acid in presence of ferric salts, with special reference to the elixir of iron, quinine, and strychnine, and elixir of iron, quinine, and strychnine phosphates. J. B. BURT (J. Amer. Pharm. Assoc., 1928, 17, 650–658).—The instability of these preparations is partly due to the oxidation of citric acid when exposed to sunlight in the presence of ferric salts; acetone and carbon dioxide are produced, and the iron is reduced to the ferrous state (cf. Benrath, A., 1918, i, 290; Plotnikow, Lehrb. d. Photochemie, 1920, 538). If air is present, ferric salts are reformed, and further quantities of citric acid are oxidised. E. W. WIGNALL.

Fluid extracts. I. Fluid extract of *Chelidonia*. A. ALBANESE and A. PEDRONI (Annali Chim. Appl., 1928, 18, 318–326).—This commercial product yields 24% of dry residue, 3% of ash (of which 42% is potassium carbonate), 11% of resinous matter, and 0.18% of alkaloids (of which 28% is β - and γ -homochelidonine). E. W. WIGNALL.

Analysis of ginger and its preparations. J. F. CLEVINGER (J. Amer. Pharm. Assoc., 1928, 17, 630–634).—Ginger rhizomes, and the pharmaceutical fluid (alcoholic) extract of ginger, contain 1.2–3% of volatile oil having d_{25}^{20} 0.875–0.885, α_D^{25} -40° to -56° , n_D^{20} 1.490–1.493, and 3.5–7.1% of non-volatile oil, soluble in ether, and containing the active constituent, iodine value 36–50, saponif. value 45–70. E. W. WIGNALL.

Conservation of the characteristics of emulsin of almonds. M. BRIDEL and (Mlle.) M. DESMAREST (J. Pharm. Chim., 1928, [viii], 8, 153–159; cf. Bridel and Arnold, A., 1921, i, 282).—The properties of the sample of emulsin prepared by acetone precipitation in 1920 and since preserved under suitable conditions have been re-examined. The properties of β -glucosidase were preserved intact; the lactase had lost about one third of its activity, and the invertase, which in 1920 hydrolysed 100% of sucrose, hydrolysed only 13.2% under similar conditions after 8 years. E. H. SHARPLES.

Commercial cholesterol. I. LIPSCHÜTZ (Chem.-Ztg., 1928, 52, 609–610).—Commercial cholesterol, which contains metacholesterol and oxycholesterol formed by the process of manufacture or by subsequent oxidation in the light, is more reactive than the pure substance; therapeutic measurements made with the commercial article are of doubtful value as it is uncertain to which constituent the action is due. F. R. ENNOS.

Use of Mitchell's ferrous tartrate reagent in studying the precipitation of alkaloids by tannin. A. E. JONES (Analyst, 1928, 53, 429–432).—When the precipitation of alkaloids is effected by addition of a measured excess of gallotannin and subsequent determination of the excess by colorimetric methods, it is found that the amount of alkaloid in the precipitate cannot thus be calculated owing to the apparent impossibility of preventing or of standardising the absorption of gallotannin by the tannate. The colorimetric method affords a means of measuring this absorption. D. G. HEWER.

Determination of adrenaline in suprarenal glands. M. PAGET and P. LOHÉAC (J. Pharm. Chim., 1928, [viii], 8, 159–169).—A modification of Bailly's colorimetric method (cf. A., 1925, ii, 248) is used. The gland is weighed, minced with three times its weight of anhydrous sodium sulphate, and the powder triturated for 5–10 min. with 5–10 c.c. of 0.1N-sulphuric acid, the volume of acid depending on the weight of the gland. After 5 min. the mixture is made up to 50 or 100 c.c. with distilled water and, after a further 15 min. with frequent shaking, it is filtered. 1 g. of sodium acetate is then dissolved in 10 c.c. of the filtrate, and a known number of drops of 5% aqueous mercuric chloride solution are added. The rose colour, which slowly appears, is compared with that given by 10 c.c. of a 0.01% solution of adrenaline treated in an exactly similar manner. Several determinations on glands after different treatments are described. Under habitual conditions of autopsy the adrenaline has disappeared after 13–14 hrs. If the glands are placed over sulphuric acid for 24 hrs. the adrenaline content is higher than that in the gland immediately after death. Examination after varying periods of cadaverisation showed that for reliable results to be obtained the gland must be removed and the powder prepared immediately after death. The increase in adrenaline after drying over sulphuric acid is ascribed to the decomposition of a labile adrenaline compound which is not detected by the above reaction, and thus for the determination of the total adrenaline, it is necessary for it to remain over sulphuric acid for 24 hrs. E. H. SHARPLES.

Determination of the iodine content of thyroid gland preparations. E. SCHULEK and A. STASIAK (Pharm. Zentr., 1928, 69, 513—514).—The method of Winterfeld and Roederer (A., 1928, 787) has the disadvantage that nitrite may be formed; following their procedure, the present authors find that traces of added nitrite can be detected after the fusion. S. I. LEVY.

Determination of camphor in alcoholic camphor solutions. E. SÜSSE (Pharm. Zentr., 1928, 69, 499—500).—An excess of salol is added to the alcoholic solution of camphor diluted with water; a yellow, oily, molecular compound of salol and camphor separates, and the excess of salol is removed and weighed. Practical details of the method are given and very accurate results are claimed. E. H. SHARPLES.

Germicidal powers and capillary activities of certain essential oils. S. RIDEAL, E. K. RIDEAL, and A. SCIVER (Perf. Ess. Oil Rec., 1928, 19, 285—304).—The connexion between germicidal power and capillary activity of 26 samples of pure essential oils has been examined. The drop numbers of the oils dissolved in B.P. paraffin at different concentrations have been determined, and also the Rideal-Walker coefficients at varying dilutions. Generally the higher the drop number of an oil the greater is its germicidal activity, and from the drop-number curves a good indication of the probable Rideal-Walker coefficient could be obtained, thus saving a number of preliminary ranging tests. Due to the chemical character of the oils, there is difficulty in relating germicidal power to chemical constitution. This is also complicated by the tendency for peroxide formation possessed by certain constituents; e.g., white camphor and eucalyptus oils form peroxides, which have considerable germicidal power. In a supplement a comprehensive table is given of the Rideal-Walker and Lancet coefficients and the physical constants of a large number of essential oils, especially eucalyptus oils, their constituents, and synthetic substances. E. H. SHARPLES.

Determination of oil in extract of lemon. Precipitation and polarimetric methods: influence of temperature and ageing on the latter. C. V. NETZ (J. Amer. Pharm. Assoc., 1928, 17, 663—668).—The percentage of oil of lemon in the commercial alcoholic extract is more accurately determined by precipitation with water than by polarimetric methods. The optical rotation is less by 8% in a 5% alcoholic solution, but generally increases on keeping.

E. W. WIGNALL.

Assay of phosphoric acid. MAURINA. **Liver of sulphur.** ROGERS.—See VII. **Insecticides.** FRANÇOIS and SEGUIN.—See XVI.

PATENTS.

Serum. Gland serum. Antitubercular serum. C. LEVENTIS (U.S.P. 1,676,995—7, 10.7.28. Appl., [A] 27.1.27, [B] 20.6.27, [C] 28.4.28).—(A) An animal is treated with an antigen, injected with human serum or with serum from an animal of different species to be protected, and the required antitoxin or antibacterial serum subsequently withdrawn. The latter may also be obtained from an animal not normally responsive to a specific disease by treating it with the specific antigen

and then injecting serum from an animal normally responsive to the disease. (B) A sexually-immature animal is castrated, implanted with a corresponding gland from a higher animal, and then injected with human serum. Serum is subsequently withdrawn from the animal. (C) The method of U.S.P. 1,676,995 (preceding) is applied. B. FULLMAN.

Rendering stable the combination of 4-dimethyl-amino-1-phenyl-2:3-dimethyl-5-pyrazolone with butyl chloral hydrate. SOC. CHIM. DES USINES DU RHÔNE, and J. ALTWEGG (B.P. 294,092, 16.5.28).—The instability of the compound is due to the liberation of traces of hydrochloric acid from the chloral, and may be obviated by the addition of calcined magnesias either during the preparation of the compound or subsequently. B. FULLMAN.

Manufacture of 4-alkylquinolines and derivatives thereof. SCHERING-KAHLBAUM A.-G. (B.P. 283,577, 30.12.27. Ger., 14.1.27).—Arylamines are condensed with alkyl β -halogenoethyl ketones in aqueous instead of non-aqueous media (cf. Blaise and Maire, A., 1908, i, 566) to give 4-alkylquinolines in improved yield. 4-Methylquinoline (from aniline, methyl β -chloroethyl ketone, and mineral acid in nitrobenzene), 6-methoxy-4-methylquinoline (from *p*-anisidine), the 8-methoxy-isomeride (from *o*-anisidine), m.p. 83°, b.p. 170°/11 mm. (picrate, m.p. 184°), 6-ethoxy-4-methylquinoline (from *p*-phenetidine), 6-nitro-4-methylquinoline (from *p*-nitroaniline), m.p. 137°, 6-chloro-4-methylquinoline (from *p*-chloroaniline), m.p. 71—72° (picrate, m.p. 222°), 4-methylquinoline-8-carboxylic acid (from anthranilic acid), m.p. 186—187°, and 4-ethylquinoline (from aniline and β -chlorodiethyl ketone; picrate, m.p. 195°) are described. C. HOLLINS.

Manufacture of derivatives of substituted quinolinecarboxylic acids. SOC. CHEM. IND. IN BASLE (B.P. 270,339, 30.4.27. Switz., 30.4.26).—A 2-chloroquinoline-4-carboxylic halide is treated first with ammonia or an amine, and the chlorine in the resulting amide is replaced by an ether or an amino-group by the action of alkali ethoxides or phenoxides, or ammonia or amines. The following compounds are described: 2-chloroquinoline-4-carboxylic dimethylamide, m.p. 114°; diethylamide, m.p. 124°; dipropylamide, m.p. 77°; diallylamide, m.p. 104°; diisobutylamide, b.p. 185°/0.015 mm.; piperidide, m.p. 140°; ethylanilide, m.p. 126°; ethylamide, m.p. 143°; benzylamide, m.p. 217°; ethylenediethyldiamide [bis-2-chloroquinoline-4-carboxyl-*NN'*-diethylethylenediamine], m.p. 74°; 2-ethoxyquinoline-4-carboxylic amide, m.p. 205°; dimethylamide, m.p. 69°; ethylamide, m.p. 152°; diethylamide, m.p. 68°; *di-n*-propylamide, m.p. 60°; diallylamide, m.p. 53°; diisobutylamide, oily; piperidide, m.p. 90°; ethylanilide, oily; benzylamide, m.p. 166°; ethylenediethyldiamide, m.p. 98°; and the following 2-substituted quinoline-4-carboxylic diethylamides: 2-methoxy-, m.p. 93°; 2-*n*-propoxy-, m.p. 61°; 2-isopropoxy-, oily; 2-allyloxy-, m.p. 33°; 2- β -phenylethoxy-, m.p. 59°; 2-cyclohexyloxy-, m.p. 63°; 2-phenoxy-, m.p. 112°; 2- β -diethylaminoethoxy-, b.p. 168—170°/0.005 mm.; 2-diethylamino-, b.p. 165°/0.02 mm. (picrate, m.p. 178°). The products are therapeutically valuable. C. HOLLINS.

Manufacture of organic tin compounds. C. T. J. VAUTIN and C. V. STEPHENS (B.P. 294,287, 22.4.27).—Methyl iodide is added to a solution of sodium stannite in aqueous alcohol, the temperature being maintained at 30–40°. After acidification, filtration, and boiling with ammonia methylstannoxyllic acid, MeSnO_2H , is precipitated. This is converted by hydriodic acid into pure methylstannic tri-iodide [methyltri-iodostannane], m.p. 84–86°, which after recrystallisation from acetic acid yields a preparation (containing a little solvent) of the same m.p. which dissolves readily in cold water. C. HOLLINS.

Testing of nicotine solutions. E. COX, Assr. to TOBACCO BY-PRODUCTS & CHEM. CORP. (U.S.P. 1,678,636, 31.7.28. Appl., 30.1.25).—To nicotine solutions (such as occur in dipping baths) after acidification, sufficient tungsten trioxide is added to combine with the organic impurities present, followed by direct titration with silicotungstic acid. H. ROYAL-DAWSON.

Preparation for the cure of foot-rot in sheep, and for external application in other animal diseases. W. EVANS (B.P. 294,734, 23.5.27).

Remedial principles of oils (U.S.P. 1,678,454).—See XII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Reduction of the latent image. LÜPPO-CRAMER (Z. wiss. Phot., 1928, 25, 129–133).—So long as there is no solvent present for the silver halide adsorbed to the silver of a negative, all silver solvents have a greater reducing action on the higher densities. This is shown to be the case with ammonium persulphate, potassium permanganate, cupric chloride, ferric chloride, potassium ferri-cyanide, mercuric chloride, and quinone. With persulphate and cupric chloride there is also fogging, although this does not occur in the case of persulphate with little-ripened emulsions. With peroxide acting on plates exposed beneath a wedge and developed after the treatment, there is at first a formation of fog and an intensification of the latent image. With increased action the latent image is reduced and fog is decreased. With sufficient action fog may be reduced to less than its original value. Similar results were obtained with persulphate. W. CLARK.

Nucleus poisoning with dyes. LÜPPO-CRAMER (Z. wiss. Phot., 1928, 25, 133–137).—Process plates were exposed, developed in metol-quinol, fixed, and washed, and then bathed in dye solutions. They were then reduced in 2% ammonium persulphate. The adsorption of the dyes retarded the reduction, the effect varying with the dye, but being independent of its being a sensitiser or desensitiser. No relation could be found between the nature of the dye and its effect. Rhodamine B completely stopped reduction by persulphate. Dyes have a similar retarding action on physical development, an effect which may account for the bad results often obtained with such development when sensitisers or desensitisers have been used. An

analogous case is the fact that the latent image on silver bromide-collodion is considerably reduced by bromide ions, whereas that on silver bromide-gelatin is not. This may be due to a similar protective action of the gelatin. W. CLARK.

Photographic diffusion-halation. R. WILDT (Z. wiss. Phot., 1928, 25, 153–184).—The properties of the photographic emulsion as a turbid medium are discussed, and an expression is evolved for the intensity distribution in diffusion-halation, which is discussed in detail. W. CLARK.

Photographic enlargement of small solid objects and the limitation of definition obtainable on gelatin plates. A. MALLOCK (Nature, 1928, 122, 239–240).

Effect of sunlight on citric acid. BURT.—See XX.

PATENTS.

Manufacture of photographic negatives and prints. L. KLAVER (B.P. 269,912, 21.4.27. Holl., 21.4.26).—A sensitive material with an easily transferable light-sensitive layer is prepared by coating the sensitive emulsion on to a temporary support consisting of a dark red or black, non-transparent, liquid-pervious paper which has been coated with a solution of casein in ammonia solution and dried. The sensitive layer is removed by damping the rear side of the support with dilute ammonia solution. J. W. GLASSETT.

Preparation of coloured reproductions by imbibition. TECHNICOLOR MOTION PICTURE CORP. (B.P. 270,279, 19.4.27. U.S., 30.4.26).—In the preparation of coloured films by imbibition printing from a dyed gelatin matrix on to a wetted gelatin film, much purer colour values and a greater range of colour contrasts are obtained if the dye solution be purified by the addition of a solution of albumin or similar amphoteric colloid, followed by coagulation of the colloid. Thus treated, the dye is imbibed much more evenly and rapidly, but in order to prevent side diffusion, which results in diffused margins, a small amount of a second dye (Metanil Yellow) of low penetrability is added. J. W. GLASSETT.

Production of hardened gelatin impression receiving surfaces [films], more especially for imbibition printing. TECHNICOLOR MOTION PICTURE CORP. (B.P. 270,280, 19.4.27. U.S., 30.4.26).—Celluloid films are coated with a gelatin solution containing potassium dichromate in the proportion of approx. 5% of the gelatin used and a small quantity of acetic acid. This coating is allowed to dry and is then subjected to heat treatment at 32–43° until a sufficient degree of hardening has been attained. J. W. GLASSETT.

XXII.—EXPLOSIVES; MATCHES.

PATENT.

Treatment of nitrocellulose [for removal of water]. HERCULES POWDER Co., Assees. of J. B. WIESEL (B.P. 293,291, 20.10.27. U.S., 1.7.27).—

Water-wet nitrocellulose is dehydrated with *sec.*-butyl alcohol. When the water has been removed, as much butyl alcohol as possible is centrifuged or pressed out, leaving 20–40% in the nitrocellulose, which keeps it moist and lessens the fire-risk. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Atmospheric pollution (Dept. Sci. Ind. Res., Rep. 13, 1928, 54 pp.).—The deposits obtained from 80 stations are tabulated and classified. A general decrease in the amount of sulphates deposited is observable—a natural result of the increased substitution of gas for coal as a source of heat. At Burnley, 2.7% of the total deposit is attributed to tar. A simple apparatus is described by means of which a sample of haze particles can be obtained for microscopical examination.

C. JEPSON.

Toxicological analysis of air, diffusion of fumes, and results of experiments at the Eiffel Tower. II. E. KOHN-ABREST (Chim. et Ind., 1928, 20, 30–36).—Four samples of air taken simultaneously at the base of the Eiffel Tower and at 57, 115, and 288 m. from the ground afforded 31, 31, 31, and 37.5 pts. of carbon dioxide and 0.0, 1.5, 1.0, and 0.0 pts. of carbon monoxide per 10,000. The increased pollution of the air at higher levels is noteworthy. F. R. ENNOS.

Ethylene oxide as fumigant. R. T. COTTON and R. C. ROARK (Ind. Eng. Chem., 1928, 20, 805).—Experiments on a small scale and in a 500 cub. ft. chamber show that ethylene oxide at the rate of 1–2 lb. per 1000 cub. ft. of space is completely toxic to a number of different kinds of insect pests of stored products. The vapour is rather more toxic than carbon disulphide, and, owing to its low b.p., the compound is effective at comparatively low temperatures (15–25°). Ethylene oxide has pronounced powers of penetration, and has no harmful effects on foodstuffs (liquid foods were not tested), clothing, furniture, or metals. It does, however, seriously affect the germination of wheat.

C. T. GIMINGHAM.

Comparative tests with certain fumigants. L. F. HOYR (Ind. Eng. Chem., 1928, 20, 835–837).—Values are given for the efficiency of a number of fumigants against various stored-product insects. Among those tested, commercial calcium cyanide at 24–27° for 24 hrs. gave a 100% kill of all insects used at 0.5 lb. per 1000 cub. ft.; chloropicrin at 0.8 lb.; "Lethane 23" at 1.5 lb.; ethylene oxide at 2 lb. Details of the effects on foodstuffs are also given. With the exception of ethylene oxide, none of the substances tested affected the germination of seeds.

C. T. GIMINGHAM.

Dialysis of putrescible liquids. O. M. URBAIN (Ind. Eng. Chem., 1928, 20, 811).—Apparatus is described by means of which the liquids, e.g., sewage, creamery waste, etc., may be dialysed without serious bacterial decomposition or oxidation occurring. The dialysing tray is enclosed in an atmosphere of nitrogen, and the dialysing water used is oxygen-free distilled water. The temperature is kept at about 0° by means of an ice jacket.

C. JEPSON.

Treatment and disposal of distillery slop by anaerobic digestion methods. S. L. NEAVE and A. M. BUSWELL (Ind. Eng. Chem., 1928, 20, 837–838).—The waste liquors from the stills during the production of industrial alcohol give rise to nuisance when diluted and allowed to ferment under aerobic conditions. If dilutions of 1:9 are inoculated with well-digested Imhoff sludge and allowed to ferment anaerobically, the waste is completely digested in 10–11 weeks with a destruction or gasification of 55% of the total solids in the slop and the production of a rapidly draining, inoffensive sludge. Dilutions less than 1:4 are too acid to permit bacterial growth. C. JEPSON.

Electrochemical polarisation process for prevention of corrosion in locomotive boilers. L. O. GUNDERSON (Ind. Eng. Chem., 1928, 20, 866–869).—The distribution of the damage done by corrosive water depends on the temperature and rate of circulation as well as on the chemical analysis. Corrosion may also occur with alkaline waters of p_H 10.5. The author has observed currents of as much as 2 amp. between the flues and the boiler still, and this factor accounts for much corrosion. It may be prevented by the addition of an arsenic salt to the water, together with the passage of a small electric current. An excess of polarised hydrogen is constantly being "plated out," and this destroys any oxygen adjacent to the metal surface. By this means pitting and grooving have been entirely prevented over a period of 4 years. C. IRWIN.

Formation of boiler scale. R. STUMPER (Chim. et Ind., 1928, 20, 10–20).—The formation and composition of boiler scale are discussed in the light of physico-chemical laws. Three types are recognisable according to their crystalline texture. With strong or rapid heating the crystals are all arranged at right angles to the surface of the metal, but if the heat flux is reduced a tangled, irregular mass of crystals is produced. The third and commonest type consists of alternate layers of the first two.

F. R. ENNOS.

PATENTS.

Prevention of boiler incrustation. W. LAZARUS (B.P. 283,517, 29.10.27. Ger., 12.1.27).—A quantity of alkali or alkali carbonate adapted to the total hardness of the water is added at the beginning of the operation, together with a larger quantity of soot, vegetable or animal carbon, or graphite, and thereafter alkali or carbonate is added in regular amounts and in shorter periods to convert calcium and magnesium salts remaining dissolved after boiling. W. G. CAREY.

Manufacture of boiler composition. H. KARPLUS and W. BACHMANN, Assrs. to E. DE HAEN A.-G. (U.S.P. 1,679,037, 31.7.28. Appl., 8.5.26. Ger., 20.11.22).—The composition, which also serves as a disinfectant, consists of a mixture of colloidal graphite and sulphite liquor, the former being one sixth of the mixture by wt.

H. ROYAL-DAWSON.

Base-exchange substances (B.P. 266,313).—See VII. **Nicotine solutions** (U.S.P. 1,678,636).—See XX.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

OCT. 12, 1928.

I.—GENERAL; PLANT; MACHINERY.

Safe heating of autoclaves. A. SANDER (Chem. Fabr., 1928, 515—516).—The direct heating of autoclaves under high pressure involves the risk of local overheating, and the use of metal or oil baths is not entirely safe. Superheated steam requires a jacket capable of resisting high pressures, whilst the use of superheated water in a coil involves structural difficulties in apparatus of cast steel. The use of superheated oil is more satisfactory if the velocity of the oil through the superheater is great enough to prevent the formation of incrustations. The author combines the use of oil and water for the heating of large autoclaves under high pressure to 200°. The water is heated by coke in a separate room to 250—300°, and its heat is then transferred to mineral oil of high flash point, the pressure of which need not exceed 10 atm. The heated oil feeds the jackets of the autoclaves. The avoidance of overheating of the oil allows its conductivity and viscosity to remain unaffected for a long period. C. IRWIN.

Spray drying. E. D. STEWART (Chem. Met. Eng., 1928, 35, 470—472).—The Douthitt Gray-Jensen spray dryer includes a conical drying chamber with central liquid spray and tangential air inlets through slots and a preliminary chamber with coarser spray. This filters the exit air and effects a preliminary concentration of the liquid. Apart from milk, this method has been successfully applied to blood products, eggs, gelatin, soap, and sodium sulphate. Vegetable flour presents difficulties owing to the viscosity of the solution, and sugar cannot be obtained as a solid unless a colloid is present. The advantage offered by the low and well-controlled temperature is principally useful for food products. C. IRWIN.

Oxygen concentration for explosion prevention [in dusts]. P. W. EDWARDS and R. W. HARRISON (Chem. Met. Eng., 1928, 35, 479—481).—The prevention of the explosion of certain dusts in air to which carbon dioxide was added was studied by the aid of a bronze bomb fitted with a cup from which dust could be blown into the enclosed air-gas mixture. Proportions of dust giving maximum pressure development on explosion were used. The lower limit for development of pressure on explosion was found to be: oat hulls 13.7% of oxygen, cork dust 14.1%, and pyrethrum flowers 15.5%. C. IRWIN.

Improved Orsat apparatus for the analysis of flue gases. K. MÜNZER (Chem. Fabr., 1928, 518—520).—The usual glass cocks are replaced by needle valves, and the scale of readings on the burette is increased by constrictions at the points where accurate readings are chiefly necessary. India-rubber is eliminated, and

the glass bulbs are very robust. The case is of metal, so as to give an apparatus durable under works' conditions. C. IRWIN.

Applicability of the analytical quartz lamp to mixtures of solid substances. F. HEIN and W. RETTER (J. pr. Chem., 1928, [ii], 119, 368—370).—Admixture of a fluorescent substance with a supposedly inert material frequently masks or even destroys the fluorescence. Thus 1 pt. of zinc oxide is not detectable by the analytical quartz lamp when mixed with 2—3 pts. of iron oxide, 5—8 pts. of cinnabar, or 20—25 pts. of chromic oxide. The fluorescence of uranyl sulphate is completely masked by 4—5 pts. of iron oxide, or 8—10 pts. of cinnabar. Anisil shows no fluorescence in presence of 60—70 pts. of iron oxide, 200—250 pts. of cinnabar, or 350—400 pts. of chromic oxide. The effect of other diluents is also recorded. It is necessary, therefore, to separate the fluorescent substance from all masking diluents before examination in the quartz lamp. C. HOLLINS.

Plasticity relationships in the technology of plastic materials and products. O. MANFRED and J. OBRIST (Z. angew. Chem., 1928, 41, 971—977; cf. A., 1927, 514; B., 1927, 563, 838).—The authors' previous work is reviewed and general data regarding the modulus of elasticity, breaking strain, etc. of cellulose products, casein products, artificial resins, rubber compositions, clays, and metallic bodies are discussed from the point of view of the directional or "stream effects." There exists a definite relationship between the degree of plasticity of the raw material and the physical properties of the moulded article. The principle of the "aggregation form" is the dominant principle in the technology of plastics. A. G. POLLARD.

Effect on heat flow through an insulating wall of modifications of shape of its isothermal boundaries. F. H. SCHOFIELD (Phil. Mag., 1928, [vii], 6, 567—592).—Mathematical.

Lubrication. BARNARD.—See II. **Pump for molten salts.** OETTINGER.—See VII. **Plasticity.** RUFF and RIEBETH.—See VIII. **Determination of colour numbers.** STOCK.—See XII.

PATENTS.

Apparatus for measuring and recording variations in temperature. W. C. HERAEUS G.M.B.H. (G.P. 448,217, 5.3.25).—A vessel closed by a mercury seal or a membrane to which is attached means for recording changes in volume of the contents, contains a gas stable at high temperatures, e.g., nitrogen, carbon dioxide, or carbon monoxide, and absorbent material, e.g., active charcoal or silica gel. L. A. COLES.

Atomising solid material. T. GOLDSCHMIDT A.-G., and V. KOHLSCHÜTTER (G.P. 439,509, 18.5.21. Addn. to G.P. 438,221; B., 1927, 463).—Superheated steam is added to the material before electrical precipitation in the process described previously. L. A. COLES.

Apparatus for mixing liquids. I. G. FARBENIND. A.-G., Assees. of H. PRILLWITZ (G.P. 448,255, 30.8.24).—Liquids are charged into a mixing vessel through inlet pipes directing the stream of liquid on to a rotating paddle agitator. L. A. COLES.

Purification of liquids. G. C. LEWIS, Assr. to DARCO SALES CORP. (U.S.P. 1,678,676, 31.7.28. Appl., 13.5.26).—The liquids are passed through a filter made by filling a ceramic container with char and burning it; the charcoal is thus activated and the walls of the container are made porous. F. G. CLARKE.

Dialysis of [putrescible] liquids. O. M. URBAIN, Assr. to TRAVERS PROCESS CORP. (U.S.P. 1,680,349, 14.8.28. Appl., 15.7.27).—A dialysing tray floated in water in a zone free from oxygen is used. F. G. CROSSE.

Apparatus for electrical precipitation of suspended particles from gaseous fluids. LODGE-COTTRELL, LTD. From METALLBANK & METALLURGISCHE GES. A.-G. (B.P. 295,890, 22.12.27. Addn. to B.P. 216,789; B., 1924, 622).—The membrane previously described (B.P. 230,841; B., 1925, 656) is attached to a hollow member insulated from the walls of the apparatus by an insulator which projects outwardly from the precipitating space. J. S. G. THOMAS.

Filter for treating air and other gases. M. BERLOWITZ (G.P. 448,891, 7.3.25).—Natural or artificial asbestos in the form of spun fibres of such length (about 1 cm.) that they can be compressed to offer the maximum resistance to the air stream is packed into filter chests. L. A. COLES.

Heat interchanger. C. V. BOYS (U.S.P. 1,680,850, 14.8.28. Appl., 24.10.24. U.K., 3.1.22).—See B.P. 195,180; B., 1923, 528 A.

Heat-treatment of substances by means of molten metal. J. S. MORGAN, Assr. to THERMAL INDUSTRIAL & CHEMICAL (T.I.C.) RES. CO., LTD. (U.S.P. 1,681,808, 21.8.28. Appl., 20.11.22. U.K., 4.1.22).—See B.P. 192,572; B., 1923, 297 A.

Drying apparatus. J. B. VERNAY (U.S.P. 1,681,690, 21.8.28. Appl., 31.10.27. Fr., 18.12.26).—See B.P. 282,432; B., 1928, 430.

Effecting the separation of subdivided materials. [Vertical separators.] R. F. GRANT, E. B. WORTHINGTON, and W. L. JACOBUS, Assrs. to SUSQUEHANNA COLLIERIES CO. (U.S.P. 1,669,820, 15.5.28. Appl., 7.9.26).—See B.P. 286,951; B., 1928, 352.

Agglomeration of pulverulent materials. H. S. SCHULTZE, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,656,572, 17.1.28. Appl., 5.5.27. Ger., 10.4.26).—See B.P. 269,209; B., 1927, 831.

Apparatus for washing and sorting coal and the like. F. BASCOUR, Assr. to SOC. ANON. CRIBLA (U.S.P. 1,681,164, 21.8.28. Appl., 20.7.26. Belg., 28.7.25).—See B.P. 256,211; B., 1927, 132.

Concentration of incrusting or corrosive solutions. P. BRINGHENTI (U.S.P. 1,682,265, 28.8.28. Appl., 15.1.26. Italy, 26.10.25).—See B.P. 254,944; B., 1926, 776.

Production of [liquefied] dry gases. G. WIETZEL and F. STOEWENER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,682,588, 28.8.28. Appl., 29.6.25. Ger., 30.6.24).—See B.P. 257,372; B., 1926, 904.

Treatment of gaseous mediums with liquids. E. M. SALERNI (U.S.P. 1,644,089, 4.10.27. Appl., 22.11.26. U.K., 21.9.26).—See B.P. 282,493; B., 1928, 176.

Compression refrigerating process and apparatus. CHICAGO PNEUMATIC TOOL CO., Assees. of R. W. DAVENPORT (B.P. 281,622, 27.10.27. U.S., 30.11.26).

Reversing absorption refrigerating apparatus. J. O. BOVING (B.P. 295,836, 31.8.27).

Composition of matter [laminated paper device] for magnetic screening etc. W. DUNBAR (B.P. 295,613, 14.5.27).

Circular kiln (U.S.P. 1,674,992).—See VIII. Distillation of solvents from solutions (B.P. 285,380).—See XII.

II.—FUEL; GAS; TAR; MINERAL OILS.

Strength [against impact] and hardness of various types of coal. D. J. W. KREULEN (Brennstoff-Chem., 1928, 9, 264—267).—Methods of carrying-out "shatter" and "rumbler" tests on coals are described, and the results from such tests on a series of coals of different rank are tabulated and plotted. In general, the hardness, as measured by either method, decreases in passing from "flaming-gas" coals through "gas" coals to "fat" coals, increasing again through "lean" coals to anthracites. Certain coals, e.g., anthracites, may appear more resistant against friction than against impact, depending on the relative hardness and friability. The bearing of the results on the liability of coals to spontaneous ignition is discussed.

A. B. MANNING.

Coal conductivity cell. E. SINKINSON (Ind. Eng. Chem., 1928, 20, 862—865).—For the measurement of the electrical resistance of coal it is necessary that it should be powdered and enclosed in a suitable cell. This may consist of a pair of wide glass tubes closed by three brass plungers (one common to both) which serve as electrodes and also for compressing the samples. One tube contains the sample, the other 1 g. of beechwood charcoal, the resistance of which, as measured on a bridge, is kept constant. A pressure of 7000 g. and a fineness of 48-mesh were used. The resistance of the brass-coal surface was compensated for. Very wide variations were found in the resistance of various anthracites, from 5×10^6 ohms down to 75 ohms, and these variations had little connexion with the analysis of the ash. The resistance of fusain was low. The boiling of coal with concentrated hydrochloric acid was found to reduce its resistance considerably.

C. IRWIN.

Carbonyl numbers of some humic acids. H. LEOPOLD (Brennstoff-Chem., 1928, 9, 215—217).—

Three preparations of humic acid, Merck's reagent, Stärke's reagent, and a preparation from quinol by Eller's method have been analysed by the phenylhydrazine method of Fuchs and Leopold (B., 1927, 315, 377), the mean carbonyl oxygen contents being 1.27, 2.31, and 2.16%, respectively, corresponding to mol. wt. of 1260, 700, and 740. These results were confirmed by Strache's method (B., 1893, 185), whilst Hoepner's method (A., 1919, ii, 434) gave carbonyl oxygen contents of 0.22, 0.40, and 0.35%, respectively.

A. E. MITCHELL.

Application of the float-and-sink method for isolating the organic constituents of carbonaceous shales. K. LUTS (Brennstoff-Chem., 1928, 9, 217—218).

—The dried shale, ground to 4900 meshes/cm.², is mixed with sodium hydroxide or calcium chloride solution (d 1.06—1.15) and the separation effected in a centrifugal machine. The results are roughly in accordance with the chemical analyses. A. E. MITCHELL.

Volatile matter and reactivity of coke. Y. OSHIMA (J. Soc. Chem. Ind. Japan, 1928, 31, 506—507).

—Determinations have been made of the moisture and volatile matter of coke under different conditions: (a) air-dried sample, (b) sample (a) exposed to the atmosphere for 24 hrs. after the determination of moisture and volatile matter, (c) sample (a) exposed to the atmosphere for 24 hrs. after preparation, and (d) sample (a) immersed in water for 24 hrs. and then air-dried. It is concluded that coke absorbs moisture which is not completely driven off at 105—110°, but is removed at a higher temperature with the so-called volatile matter. Consequently, volatile matter of coke differs from that of coal in that it is mostly moisture which is absorbed or occluded, due to the surface structure. It follows, therefore, that the reactivity of coke is influenced by surface conditions, porosity, and the state of the carbon—conditions which govern the absorption—rather than by the content of so-called volatile matter.

K. KASHIMA.

Countercurrent use of decolorising carbons.

M. T. SANDERS (Ind. Eng. Chem., 1928, 20, 791—794).—A mathematical treatment, based on Freundlich's adsorption equation, of the two- and three-step countercurrent processes of using decolorising carbons. Graphical methods are illustrated and a nomogram is given for determining the relative amounts of decolorising carbon required in alternative methods of application.

H. S. GARLICK.

Carbonisation in vertical retorts. J. L. HYSLOP (Gas J., 1928, 183, 447—449).—On account of the wide range of conditions which can be obtained in vertical retort settings, careful control of operation is necessary. Steaming of retorts, fuel consumption, waste-heat boilers, sizing of coal, and cost of gas production are discussed, and figures are given relative to the effect on the quality of producer gas of differences in methods of ash removal from the grate, and to the influence of the capacity of the foul main on the pressure conditions maintained in the retort.

R. H. GRIFFITH.

Influence of water content of coal and coke on retort performance and retort guarantees. E. DUBOIS (Gas- u. Wasserfach, 1928, 71, 793—798).—

Increased moisture content of the coal charged to a gas retort results in a very considerable decrease in the throughput and in the gas yield. Calculation shows that the effect on the latter is double that on the former, the actual values being dependent on the heat of coking of the coal. *E.g.*, an increase in moisture content from 2% to 12%, for a coal with a heat of coking of 350 g.-cal./g., lowers the throughput by 10.2%. The consequent increased fuel consumption with high-moisture coals is greater for settings with self-contained generators than for those heated by gas from an external source. The water content of the coke used for firing the former type of setting has an injurious effect not only on the fuel consumption, but also on the combustion temperatures. The relation between fuel consumption and moisture content of the coke is discussed.

A. B. MANNING.

Explosion limits of technical gas mixtures.

K. BUNTE and A. STEDING (Gas- u. Wasserfach, 1928, 71, 821—822).—The explosive limits of a gas-air mixture are determined by the points at which the ignition velocity becomes zero. The apparatus of Eitner has been used to measure these limits for mixtures of coal gas with carbon dioxide, nitrogen, and water-gas; the addition of carbon dioxide or nitrogen raises the minimum concentration of gas necessary to cause explosion and narrows the range over which this can occur. Admixture with water-gas affects the lower limit to a small extent only, but increases the explosive range very considerably.

R. H. GRIFFITH.

Experimental oil-shale plant of the Bureau of Mines.

M. J. GAVIN (Ind. Eng. Chem., 20, 1928, 784—791).—A description of the mine and retorting plant at Rulison, Colorado. The shales range in richness from a few gals. to over 65 gals. of oil per ton. A continuous vertical section of shale yields 30.47 gals. of oil per ton. The oil-producing equipment comprises two retorts—a standard commercial size Pumphreyston and a 25-ton capacity N-T-U retort. With a throughput of 7.5—8 tons per day the former was yielding an oil recovery of 93.2—100.1% on a shale assaying 29 gals. per ton. The gas yield varied inversely with the throughput rate and amount of steam supplied, the retort being practically self-supporting as regards fuel. The N-T-U retort gave oil recoveries ranging from 36 to 94% of the assay values of the shales. Some difficulty was experienced owing to the formation of oil fog. The oils produced in the two retorts are essentially similar except that those from the N-T-U retort are uniformly heavier and contain less of the more volatile fractions.

H. S. GARLICK.

Theory of the formation of petroleum.

G. STADNIKOV and E. IVANOVSKI (Brennstoff-Chem., 1928, 9, 261—264; cf. B., 1928, 700).—The fatty acids from linseed oil have been decomposed by heating at 400° in the presence of an iron catalyst, yielding thereby about 76% of a product with an acid value of 0.5 and an iodine value of 153. The steam distillate from this product consisted almost entirely of a mixture of hydrocarbons, mainly unsaturated but containing also some paraffin and aromatic hydrocarbons and traces of naphthenes. The unsaturated hydrocarbons were

aliphatic in nature, yielding simple fatty acids on oxidation with alkaline permanganate. The residue from the steam-distillation was fractionated under reduced pressure; in addition to hydrocarbons these fractions contained some oxygen compounds and had acid values in the region of 2. It is concluded from the close resemblance between these products and those obtained by the low-temperature distillation of Tscheremchovski boghead coal that the latter consists of polymerised fatty acids.

A. B. MANNING.

Composition of petroleum (kerosene and other) fractions. J. A. CARPENTER (J. Inst. Petroleum Tech., 1928, 14, 446—476).—Aniline points of paraffins and of naphthenes rise with mol. wt., but those of naphthenes are lower than those of corresponding paraffins. Aniline points of aromatic hydrocarbons are from -20° to 0° . The values for unsaturated hydrocarbons are intermediate between those for aromatic hydrocarbons and naphthenes, and vary greatly with constitution. Whilst the presence of naphthenes and more particularly of aromatic hydrocarbons may be followed fairly accurately by means of aniline point depression, unsaturated hydrocarbons produce too irregular an effect. Aromatic hydrocarbons are isolated by Armstrong's method. Solvent extraction at low temperatures produces a concentration of aromatic hydrocarbons, but pure hydrocarbons cannot be isolated by this method alone. Paraffins from $C_{20}H_{42}$ to $C_{33}H_{68}$ and $C_{57}H_{118}$ are prepared from waxes, which are distilled under reduced pressure or with steam, the resulting fractions being then recrystallised from solvents or by fractional cooling. Paraffins from $C_{13}H_{28}$ to $C_{19}H_{40}$ are isolated by fractional distillation after removal of aromatic hydrocarbons. The narrow fractions are then submitted to drastic sulphonation or nitration, or to solvent extraction, or a combination of these methods, and are finally purified by fractional freezing. Lower paraffins are prepared by similar methods excluding freezing. The selective solvent action of aniline for different classes of hydrocarbons is accentuated by the presence of water. The following have been identified in a fraction of Burmah crude oil to 155° : *n*-butane, isobutane, *n*-pentane, isopentane β - or γ -methylpentane, *n*-hexane, methylcyclopentane, dimethylpentanes, cyclohexane, impure β -methyl- β -ethylbutane, either or both of the dimethylcyclopentanes, methylcyclohexane, *n*-heptane and (probably) $\beta\beta\gamma\gamma$ -tetramethylbutane, dimethylhexanes, 1:3-dimethylcyclohexane, a fraction of b.p. 135 — 140° , giving trinitromesitylene and trinitro- ϕ -cumene on vigorous nitration, a paraffin, d 0.725, contained in the fraction of b.p. 152.5 — 155° , and hexahydrocumene. Burmah wax, m.p. 58° , when cracked at 418° and 140 lb./sq. in. yielded 90% of pressure distillate. The portion of b.p. up to 175° contained 19% of olefines, 14.5% of aromatic hydrocarbons, a small proportion of naphthenes, and the remainder paraffins. The kerosene obtained by distillation with steam had 3% of olefines and 12% of aromatic hydrocarbons. B.p., m.p., and aniline point values are given for 24 paraffins up to $C_{60}H_{122}$ isolated from petroleum. With the exception of the lowest members of the spirit range, the values correspond with those for side-chain hydrocarbons. Aniline points are given for various naphthenes

isolated from petroleum and aniline points, d , and n_D^{20} are given for fractions, b.p. 125 — 300° , from various crude oils. Tentative rules are suggested for determining the classes of hydrocarbons in kerosene. W. S. NORRIS.

Comparison of Soviet and American kerosenes. S. NAMETKIN (Neft. Choz., 1927, 13, 328—331).—Soviet kerosene has a wider boiling range than American; 70—75% is volatile at 100 — 110° , and the final b.p. is higher. Soviet kerosene has more heavy gasoline and light solar oil fractions, a high (5—10%) content of unsaturated compounds, and a negligible sulphur content.

CHEMICAL ABSTRACTS.

Tube-still distillation. C. H. S. EDMONDS (J. Inst. Petroleum Tech., 1928, 14, 388—393).—The "single-flash" or "once-through" method of tube-still distillation has advantages when a topping or stripping operation is to be performed, whilst the "step-up" system is particularly suited for deep cutting into the crude oil, since the later stage of evaporation may be performed under reduced pressure. The single-flash system, which takes full advantage of the partial-pressure effect of the light components, may, however, be adapted to give both light and heavy distillates in one operation. The value of tube-still distillation units depends on the efficiency with which heat is transferred to the oil and later removed from the oil. The danger of local overheating, with consequent decomposition of the oil, is minimised by the use of a tube furnace in which advantage is taken of radiation. This permits of greater furnace efficiency. The fractionating tower must be flexible and easily controlled, and should give specified products without redistillation. There is a distinct trend towards the use of bubble-tray towers. Definite heat control is generally effected by pumping back reflux into the tower; for this purpose the lightest cut yielded by the crude oil is usually employed. Additional reflux at intermediate points in the tower is provided by the inclusion of "reboiling" or "stripping" sections. Economy is effected by the use of heat exchangers, as much as 40% of the total heat liberated being recovered.

W. S. NORRIS.

Sulphur toleration in gasoline. G. EGLOFF and C. D. LOWRY, JUN. (Ind. Eng. Chem., 1928, 20, 839—843).—Refining methods for the production of gasoline with a maximum sulphur content of 0.1% involve polymerisation and destruction of unsaturated compounds desirable for their high anti-knock value. Corrosion is exclusively a cold-weather problem, but even in winter it does not occur in engines in constant use, but only in those where, through condensation, water collects in the crank-case. Analysis of statistics shows that more than two thirds of the total consumption of gasoline are used in warm weather when there is no danger of corrosion. It would therefore be advantageous to distribute gasoline without regard for sulphur content in the summer months, and to reduce the sulphur in addition to increasing the volatility when cold weather conditions prevail.

H. S. GARLICK.

Determination of oxidisability of transformer oils. N. BUTKOV (Neft. Choz., 1927, 13, 332—333).—The oil is contained in a glass tube immersed in Wood's metal at 150° in an autoclave, and oxygen is led in at

4–5 litres/hr. The oil is then treated with sulphuric acid, and the acidity, formation of sludge, and saponification are determined. CHEMICAL ABSTRACTS.

Lubrication [and lubricating oils]. D. P. BARNARD, IV (Ind. Eng. Chem., 20, 1928, 843–846; cf. Parsons and Taylor, B., 1926, 620).—Comparison of viscosities in a Saybolt viscosimeter under varying heads at low temperatures showed that the deviation in the case of asphalt-base oils from the curve for a fluid obeying the ordinary laws of viscous flow was not great. With other oils the flow increased more rapidly than in proportion to the pressure. The departure from the ordinary laws of viscous flow is typical for different classes of oils, and due to the presence of a colloidal structure which becomes more pronounced as the temperature is lowered. From comparison of the viscosity as predicted by extrapolation on a Herschel chart, and that actually determined for several conditions of shearing stress, the best agreement was obtained under a pressure drop of 200 lb./in.² By plotting the cranking torque against the apparent oil viscosity it was shown that cranking effort is determined by oil viscosity corresponding to fairly high shearing stresses. Any deviation could not be correlated with any pour-test effect, which appeared to be unconnected with ease of starting. The cranking effect characteristic of a motor oil can be estimated with fair reliability by means of a Herschel diagram, but may be modified by excessive pour tests. The establishment of circulation depends entirely on the pour test, and is unaffected by viscosity characteristics. For use in automotive work an oil should possess a low temperature coefficient of viscosity and low pour test. At lower temperatures all oils exhibit plastic characteristics and do not follow the laws of viscous flow. Where deviation is great the oil is at a disadvantage owing to poor circulation characteristics. Low temperature coefficient of viscosity is of more fundamental importance at both low and high temperatures, and the effect of the pour test can be minimised by suitable enlargement of pump inlets. H. S. GARLICK.

Direct current conductivity of insulating oils. D. H. BLACK (Phil. Mag., 1928, [vii], 6, 369–384).—The diminution in current with time when a *P.D.* is applied across a liquid dielectric is attributed to the building up of a "contact resistance" at one or both of the electrodes which tends to disappear at a rate proportional to its magnitude. Experiments with films of oils in the paraffin series are cited in support of this theory. The final steady resistance is a linear function of the current strength except in freshly prepared films. Moisture seems to reduce the value of the contact resistance without materially changing the true resistance of the oil. S. K. TWEEDY.

Explosion of dusts. EDWARDS and HARRISON. **Analysis of flue gases.** MÜNZER.—See I.

PATENTS.

Plants for washing coal or other minerals. A. FRANCE (B.P. 285,864 and 286,281, 21.9.27. Belg., [A] 24.2.27, [B] 4.3.27).—(A) A coal or mineral washing plant, depending on the separation of particles of different density as they are carried along in an alluvium-

forming stream inside a horizontal or slightly inclined launder in which slots are provided for the reception of the denser material, has the launder so shaped as to aid in the separation. Thus the cross-section gradually narrows on approaching a slot from upstream, remains constant in width from just above to just below the slot, and thereafter widens again. (B) The launder is provided with movable false sides so that the shape of the cross-section in the neighbourhood of the slots can be adjusted to give the best separation with the particular material under treatment. A. B. MANNING.

Regenerative coke ovens. N. V. SILICA EN OVENBOUW MIJ., Assees. of C. OTTO & Co., GES.M.B.H. (B.P. 286,234, 28.2.28. Ger., 28.2.27. Addn. to B.P. 240,167; B., 1925, 981).—An oven of the kind described in the main patent has gas-distributing ducts beneath the soles of the oven chambers for supplying strong gas to the heating flues, so arranged that the ducts beneath each chamber are simultaneously either in communication with or disconnected from the strong-gas main. If desired, the ducts may be combined into a single duct beneath each chamber. A. B. MANNING.

Coking retort ovens. [A, B] J. BECKER, and [C] J. VAN ACKEREN, Assrs. to KOPPERS Co. (U.S.P. 1,678,801–3, 31.7.28. Appl., [A] 28.1.21, [B] 14.3.21, [C] 16.5.21).—(A) The heating walls between the coking chambers of a coke oven are constituted of two parallel series of vertical combustion flues, the individual flues of one series being connected with the corresponding flues of the other series of the same heating wall. Individually regulatable and reversible regenerators are connected in pairs with each series of combustion flues. (B) The heating walls are similarly constituted, but tapered horizontal flues in each heating wall connect both series of combustion flues, whilst the horizontal flues of each heating wall are connected with the corresponding horizontal flues of an adjacent heating wall. Individually regulatable and reversible, cross-wise, extending regenerators are divided into groups of three; two of each group are connected, one with the flues of one series and the other with the flues of the other series of an adjacent heating wall; the third of each group is connected with both flue series of the adjacent wall. (C) The heating walls of the coking chambers are constituted of vertical combustion flues disposed into two groups, the flues of one group alternating with the flues of the other, and one group operating for inflow while the other operates for outflow. Two series of regenerators are provided, one connected with the groups of flues and the other connected with individual flues, and are arranged so that both series may be used for conveying air to the flues, or one for conveying air and one for gas. A. B. MANNING.

Fuel-distilling apparatus. F. E. HOBSON and J. F. SHELTON (U.S.P. 1,672,860, 5.6.28. Appl., 6.4.25).—The apparatus comprises a long, vertical, cylindrical retort with a centrally-disposed gas-collecting flue between which and the walls of the retort is arranged a vertical, cylindrical, hanging curtain provided with louvres throughout its length. The curtain is placed close to the walls of the retort, and may be moved vertically so as to cause the charge in the retort to fall towards

the bottom and to prevent it from adhering to the walls during carbonisation. A. R. POWELL.

Apparatus for distilling lignite, peat, coal, etc. E. MARCOTTE and H. BREVILLE (F.P. 625,276, 6.3.26).—The retorts can be constantly charged and discharged by removing the base, which is provided with duplicate housing and steam apparatus. L. A. COLES.

Manufacture of carbon from carbon monoxide. CASSEL CYANIDE CO., LTD., and T. EWAN (B.P. 294,759, 5.7.27 and 13.4.28).—Carbon monoxide, preferably containing a small amount of iron carbonyl, is passed over a massive catalyst, *e.g.*, pure sheet iron, at 300–600°, the exact temperature chosen depending on the physical properties of the carbon required. The carbon produced is removed mechanically by scraping, and may be transferred to a cooler zone (60–70°), being moved counter-current to the carbon monoxide so that the latter is brought into contact with the carbon already formed before entering the reaction zone. In order to reduce the amount of iron in the product the detached carbon may be agitated for some time in the high-temperature zone. A. B. MANNING.

Production of carbon black from mixtures of acetylene and other hydrocarbons. L. R. CHURCHILL, ASSR. to GOODYEAR TIRE & RUBBER CO. (U.S.P. 1,673,496, 12.6.28. Appl., 11.7.23).—Carbon black of quality required for use in rubber is obtained by introducing a mixture of methane and air into a cooled explosion chamber, adding an almost equal volume of acetylene, the whole being at about 75 lb./in.², and detonating the acetylene with an electric spark before it has diffused through the chamber. C. HOLLINS.

Destructive hydrogenation of carbonaceous materials. I. G. FARBERIND. A.-G. (B.P. 275,662, 8.8.27. Ger., 7.8.26).—The reaction vessel and other parts of the apparatus which come into contact with the hot organic reacting materials in hydrogenation processes carried out under high pressures are made of, or coated with, materials, *e.g.*, alloys containing one or more of the metals nickel, cobalt, molybdenum, tungsten, etc., or of the metalloids boron, arsenic, antimony, silicon, etc., which do not give rise to the formation of methane or to the deposition of carbon. Copper or its alloys may be used if the reacting materials are free from sulphur. Metals or alloys prepared by fusion *in vacuo* and free from impurities at the boundaries of the crystal grains are particularly suitable for the purpose. (Cf. B.P. 268,796; B., 1928, 663.) A. B. MANNING.

Heating carbonaceous materials. I. G. FARBERIND. A.-G. (B.P. 279,825, 13.10.27. Ger., 28.10.26).—Liquid hydrocarbons or carbonaceous pastes which are to be subjected to destructive hydrogenation or other process involving high temperatures are preheated in vessels or tubes the walls of which act as electrical resistances and are traversed by a heating current. Iron pipes may be used for the purpose up to about 400°, but if higher temperatures are used the pipes should be constructed of aluminium or chromium-nickel steel in order to prevent carbon deposition or methane formation. A. B. MANNING.

Gas producers and plant in connexion therewith. R. M. A. E. CEZANNE, and SOC. D'APPL. DU GAZ AUX MOTEURS "S.A.G.A.M." (B.P. 294,773, 3.8.27).—A down-draught gas producer, designed for use on motor vehicles, is described. A. B. MANNING.

Production of reducing gases. A./S. NORSK STAAL (ELEKTRISK-GAS-REDUKTION), and E. EDWIN (B.P. 294,838, 20.12.27).—Gases containing carbon dioxide and water vapour, heated by means of a high-tension electric arc, are passed through a layer of solid carbonaceous material rendered incandescent by the sensible heat of the gases, and are thereby converted into carbon monoxide and hydrogen. The temperature of the gases entering the layer is so high (at least 1400°) that their sensible heat not only covers the heat consumption of the endothermic reactions, but also effects complete fusion of the slag. A. B. MANNING.

Treatment of hydrocarbons with electric arcs. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 294,494, 24.1.27).—Hydrocarbon gases or vapours are passed through an arc formed between a central electrode, insulated except at the end, and a surrounding, tubular, counter-electrode. A movable insulating mantle between the electrodes permits adjustment of the length of the arc to a value most suitable for the reaction. In this way increased yields of acetylene from methane and of hydrocyanic acid from gas mixtures containing methane and nitrogen are obtained. A. B. MANNING.

Manufacture of mineral oils from peat, lignite, coal, and other bituminous materials. C. W. M. BEROETS (B.P. 270,349, 2.5.27. Dutch East Indies, 1.5.26).—The starting materials are moistened with salt water, heated to 50°, and fermented by the micro-organisms normally present, under favourable conditions of time, temperature, air supply, and moisture content. A. B. MANNING.

Production of liquid and gaseous hydrocarbons of low b.p. from mineral and other oils, tars, etc. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 294,557, 21.4.27).—The vaporised oils are cracked by being passed, preferably at a high velocity, over a heating device arranged in a single plane, and of small linear dimensions in the direction of flow of the vapours. The device, which may consist of a length of pipe arranged in the form of a grate and heated internally, or of rods of metal, graphite, etc. heated electrically, is maintained at 500–700°. It is advantageous to treat the device, either intermittently or continuously, with a blast of air, hydrogen, or gases yielding hydrogen. A. B. MANNING.

Conversion of hydrocarbons of high b.p. into compounds of lower b.p. I. G. FARBERIND. A.-G. (B.P. 270,314, 28.4.27. Ger., 1.5.26).—Cracking of heavy hydrocarbon materials (*e.g.*, producer tar or crude Panuco petroleum) is carried out in the absence of metals (cf. B.P. 268,796; B., 1928, 663), but in the presence of such non-metallic catalysts as do not give rise to the deposition of carbon. Suitable catalysts are oxygen compounds of the heavy metals of the third to sixth groups of the periodic system or of iron, nickel, or cobalt (*e.g.*, molybdic acid or an artificial zeolite for the inter-changeable alkali metal of which cobalt or uranium has

been substituted). The process may be conducted under ordinary, reduced, or increased pressure; inert or reducing gases or steam may be passed through the apparatus during the process. W. S. NORRIS.

Distilling oil shale and providing a raw material for manufacture of hydraulic cement. O. TETENS, Assr. to RECORD CEMENT-IND. G.M.B.H. (U.S.P. 1,678,751, 31.7.28. Appl., 19.9.24. Ger., 14.7.24).—Columns of a mixture of shale and calcareous material are subjected to downward combustion, the columns being replenished from above and air entering at the bottom. The combustion zone is cooled externally by water, which is thus converted into steam. Oil is distilled from the top of the columns and a cement mix is discharged at the bottom. F. G. CLARKE.

Distillation of hydrocarbons. C. ARNOLD. From STANDARD DEVELOPMENT CO. (B.P. 295,142, 8.9.27).—In apparatus of the type in which oil undergoes heat-treatment in a number of series-connected stills or receptacles, transference of the oil is effected by connecting adjacent vessels by means of a pipe in which is inserted a fluid injector, both down-flow and up-flow sections of the pipe being of substantial length. The use of steam in the injector effects distillation and transference of the oil simultaneously. W. S. NORRIS.

Production of demulsifying agents for refining petroleum oil. E. D. GRAY, Assr. to STANDARD OIL CO. OF CALIFORNIA (U.S.P. 1,673,045, 12.6.28. Appl., 4.10.23).—A viscous petroleum oil, preferably after a preliminary treatment with sulphuric acid to remove tar-forming constituents, is treated with oleum and the acid sludge produced is added to a light or medium lubricating oil, which extracts from the sludge a substance having demulsifying properties. The extract (if desired, after a sulphuric acid treatment and removal of acid sludge) is made nearly neutral, decanted from heavy sulphonated compounds, and then completely neutralised; the aqueous liquor now contains the demulsifier, suitable for the "brightening" of heavy motor oils and steam-cylinder oils. C. HOLLINS.

Manufacture of motor fuels. I. G. FARBERIND. A.-G. (B.P. 281,247, 3.11.27. Ger., 26.11.26).—"Anti-knocking" motor fuels can be produced by the hydrogenation of bituminous coals if the bitumen in the original coal, or the primary conversion products of the bitumen in the partially hydrogenated material, is removed by extraction with benzene. A. B. MANNING.

Low-temperature fuel distillation. E. G. WEEKS, Assr. to MERZ & MCLENNAN (U.S.P. 1,681,450, 21.8.28. Appl., 15.1.24. U.K., 3.10.23).—See B.P. 223,387; B., 1924, 1004.

Gas producer. G. H. BENTLEY and E. G. APPLEBY (U.S.P. 1,679,645, 7.8.28. Appl., 20.5.25. U.K., 22.5.24).—See B.P. 227,346; B., 1925, 197.

Apparatus for manufacture of mixed gas. M. W. TRAVERS and F. W. CLARK, Assrs. to REGENERATIVE COAL GASIFICATION SYSTEM, LTD. (U.S.P. 1,681,313, 21.8.28. Appl., 12.2.26. U.K., 29.6.23).—See B.P. 210,356; B., 1924, 244.

Preparing artificial petroleum from rubber scrap or vulcanised rubber waste. H. NISHIDA and

K. SHIMADA (U.S.P. 1,680,908, 14.8.28. Appl., 28.1.27. Japan, 24.12.25).—See B.P. 282,565; B., 1928, 253.

Fractional extraction of petroleum hydrocarbons with alcohol. T. A. WERKENTHIN, Assr. to SOLAR REFINING CO. (U.S.P. 1,680,352, 14.8.28. Appl., 19.11.24).—See B.P. 254,784; B., 1926, 863.

[Motor] fuel. H. S. REID, Assr. to CANADIAN ELECTRO PRODUCTS CO., LTD. (U.S.P. 1,680,392, 14.8.28. Appl., 30.3.25).—See B.P. 249,348; B., 1926, 430.

Feeding devices for coal pulverising mills. E. VOGT and L. KIRCHHOFF (B.P. 295,899, 24.1.28).

Means for testing gases of combustion, particularly adapted for boiler supervising plant. SIEMENS & HALSKE A.-G. (B.P. 271,841, 22.4.27. Ger., 26.5.26. Addn. to B.P. 258,226).

Valves for use with gas purifiers. H. H. HOLLIS (B.P. 295,269, 8.4.27).

Lead tetraethyl (B.P. 279,106). **Hydrogen** (B.P. 271,483).—See VII. **Paving compositions** (B.P. 293,830).—See IX.

III.—ORGANIC INTERMEDIATES.

Preparation of xyleneols from technical xylidines and ethylphenols from benzene. H. BRÜCKNER (Z. angew. Chem., 1928, 41, 955—956).—*m*-4-Xylidine and *p*-xylidine are separated successively as acetates from technical xylidines (Birukoff, B., 1887, 541), the *m*-2-, *o*-3-, and *o*-4-isomerides by means of sulphuric acid (Busch, A., 1899, i, 496), each base being purified as formyl derivative. Waste xylidines, from which most of the *m*-4- and all the *p*-xylidine had been removed, gave 25% of *m*-4-, 21% of *m*-2-, 27% of *o*-3-, and 19% of *o*-4-xylidine. *m*-5-Xyleneol is best prepared from *m*-4-xylidine by acetylation, nitration, hydrolysis, and diazotisation (Haller, Adams, and Wherry, A., 1920, i, 670), followed by treatment of the diazo-compound with sodium zincate, reduction of the 5-nitro-*m*-xylene so formed, diazotisation, and boiling with water. Ethylphenols are conveniently obtained by ethylating benzene with ethylene in presence of aluminium chloride at 75° (Milligan and Reid, B., 1922, 245A), followed by nitration, reduction, diazotisation, and boiling with water. C. HOLLINS.

Cresol soap. KOGAN.—See XII. **2-Phenylquinoline-4-carboxylic acid.** CHEMNITUS.—See XX.

PATENTS.

Synthesis of alcohols. COMP. DE BÉTHUNE (B.P. 274,492, 15.7.27. Fr., 17.7.26).—As catalysts in the hydrogenation of carbon monoxide, especially in the production of methyl alcohol, formates of metals (which give oxides not reduced by hydrogen or carbon monoxide under the reaction conditions) are employed; *e.g.*, zinc formate or, better, a mixture of zinc and chromium formates, is deposited on active carbon by evaporation of an aqueous solution, and the catalyst is used at 300° under 800 kg./cm.² pressure. C. HOLLINS.

Purification of *N*-alkylcarbazoles. F. S. MORTIMER and R. W. HESS, Assrs. to NAT. ANILINE & CHEM. CO., INC. (U.S.P. 1,674,216, 19.6.28. Appl., 17.6.25).—

N-Ethylcarbazole is extracted from the crude product containing carbazole, anthracene, and phenanthrene, by 83–84% sulphuric acid at 0–10°. Carbazole is less soluble under these conditions in presence of an equal amount of anthracene. C. HOLLINS.

Purification of crude aromatic hydrocarbons. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,674,472, 19.6.28. Appl., 12.10.27).—The crude hydrocarbon fraction is treated with concentrated sulphuric acid, and at the same time with sufficient chlorine to chlorinate the non-aromatic impurities. Treatment with chlorosulphonic acid is also claimed as a convenient combination of sulphuric acid treatment and chlorination. C. HOLLINS.

Production of ester mixtures. W. CLAASEN (B.P. 294,947, 2.2.27. Addn. to B.P. 250,910; B., 1926, 769).—The process of the prior patent is applied to phenol, and leads to technically useful mixtures of adipic, glutaric, and succinic esters. C. HOLLINS.

Manufacture of alkylated phenols and their hydrogenated products. CHEM. FABR. AUF ACTIEN VORM. E. SCHERING (B.P. 254,753, 5.7.26. Ger., 4.7.25).—Catalytic hydrogenation of the condensation products of ketones with phenols yields alkylated phenols or cyclohexanols according to the activity of the catalyst, which may be adjusted by the addition of bismuth for retardation or of manganese for activation. 4:4'-Dihydroxy- $\beta\beta$ -diphenylpropane, treated with hydrogen at 10–20 atm. in presence of nickel catalyst containing 3–10% Bi at 160°, yields *p*-hydroxycumene and phenol. Similarly the product from *o*-cresol and acetone yields 6-hydroxy-*m*-cymene and *o*-cresol. With a nickel catalyst containing 1–8% Mn the corresponding cyclohexanols are obtained. C. HOLLINS.

Purification of *p*-nitroaniline. A. MILLER, Assr. to TOWER MANUF. Co., INC. (U.S.P. 1,673,154, 12.6.28. Appl., 14.7.25. Renewed 25.10.27).—*p*-Nitroaniline made from *p*-chloronitrobenzene is freed from impurities which produce a flocculent precipitate on diazotisation by treatment with mild reducing agents (sodium sulphide or dilute stannous chloride) in regulated amount. C. HOLLINS.

Chlorination [of naphthalene]. S. BROWN, Assr. to HALOWAX CORP. (U.S.P. 1,672,878, 12.6.28. Appl., 5.1.24).—Chlorine is aspirated through molten naphthalene in a series of vessels maintained at temperatures below the b.p. of naphthalene at the reduced pressure. The products are wax-like substances. C. HOLLINS.

Manufacture of condensation products from naphthalene or naphthalene derivatives and ethylene. I. G. FARBENIND. A.-G. (B.P. 265,601 and Addn. B.P. 273,665 [A] 3.2.27, [B] 5.5.27. Ger., [A] 4.2.26, [B] 30.6.26).—Naphthalene or tetrahydronaphthalene is treated at 100–200° with (a) ethylene or (b) other olefines or olefine mixtures in presence of aluminium chloride or ferric chloride to give ethyl- and higher alkylated naphthalenes or corresponding hydrogenated compounds. C. HOLLINS.

Production of benzanthrone derivatives. I. B. ANDERSON, R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 295,213, 2.2.27).—4-Amino-3-hydroxy-

1-*p*-amino-*o*-carboxyphenylnaphthalene (or its alkylation products), obtained from *m*-aminobenzoic acid — β -naphthol by reduction and benzidine transformation, is converted by cold 10% sodium hydroxide solution into 3:9-diamino-2-hydroxy- or alkoxy-benzanthrone, together with 9-amino-2:3-oxido-benzanthrone produced by elimination of ammonia or alkylamine. C. HOLLINS.

Manufacture of acetic acid and acetates. P. A. and H. G. SMITH, Assrs. to SYNTHETIC AMMONIA & NITRATES, LTD. (U.S.P. 1,679,994, 7.8.28. Appl., 10.3.27. U.K., 13.3.26).—See B.P. 271,589; B., 1927, 571.

Preparation of ethylidene diacetate. M. E. BOUVIER and L. HUGONOT, Assrs. to SOC. CHIM. DES USINES DU RHÔNE (U.S.P. 1,680,760, 14.8.28. Appl., 18.8.25. Fr., 13.6.25).—See B.P. 252,632; B., 1926, 692.

Manufacture of sulphonic derivatives of aralkylated unsaturated fatty acids. A. THAUSS, G. MAUTHE, and A. GÜNTHER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,667,225–6, 24.4.28. Appl., [A] 21.12.26, [B] 17.3.27. Ger., [A] 22.12.25, [B] 24.4.26).—See B.P. 286,796; B., 1928, 360.

Manufacture of [α]-hydroxy-acid esters. H. W. MATHESON and K. G. BLAIR, Assrs. to CANADIAN ELECTRO PRODUCTS Co. (U.S.P. 1,682,347, 28.8.28. Appl., 11.1.26).—See B.P. 264,143; B., 1927, 796.

Manufacture of side-chain [polynuclear] aromatic compounds. F. GÜNTHER, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,670,505, 22.5.28. Appl., 2.3.27. Ger., 3.3.26).—See B.P. 267,132; B., 1928, 597.

Manufacture of chlorinated hydroaromatic products containing nitrogen. T. VOLTZ, Assr. to DURAND & HUGUENIN SOC. ANON. (U.S.P. 1,679,998, 7.8.28. Appl., 24.2.23. Ger., 23.2.22).—See B.P. 193,843; B., 1924, 10.

Manufacture of new indophenols and leuco-indophenols, and new dyes therefrom. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 286,005, 27.11.26).—See G.P. 443,685; B., 1928, 225.

Purification of naphthalene. G. SCHROETER (U.S.P. 1,680,070, 7.8.28. Appl., 30.3.25. Ger., 13.5.16).—See G.P. 324,863; B., 1921, 253 A.

Preparation of 2:3-dichloronitro-1:4-naphthoquinone. J. STOCK, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,681,599, 21.8.28. Appl., 30.11.27. Ger., 27.3.25).—See B.P. 288,927; B., 1928, 441.

Anthraquinone-nitrosoamine compound. H. TESCHE and A. JOB, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,643,428, 27.9.27. Appl., 12.10.25. Ger., 24.10.24).—See G.P. 442,312; B., 1928, 685.

Acetic acid (B.P. 295,238).—See VII.

IV.—DYESTUFFS.

PATENTS.

Manufacture of vat dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 295,239, 4.5.27).—5-, 6-, and 7-Methylindole-2:2'-thionaphthen-indigos are dibrominated in sulphuric acid at 10–40° to give bright violet vat dyes. C. HOLLINS.

Production of grey to black vat dyes. L. B. HOLLIDAY & Co., LTD., and C. SHAW (B.P. 295,506, 28.7.27).—Dibenzanthrone is oxidised in sulphuric acid with chlorate or permanganate (cf. B.P. 277,125; B., 1927, 837). C. HOLLINS.

Manufacture of azo dyes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 295,237, 4.5.27).—Bluish-violet to blue monoazo dyes, suitable for acetate silk dyeing, are obtained by coupling diazotised picramide with aralkylanilinesulphonic acids, e.g., sulphobenzyl-ethyl-*m*-toluidine (reddish-blue), sulphobenzylmethyl-aniline (bluish-violet), sulphobenzyl-*o*-toluidine, or benzylaniline-*o*-sulphonic acid. 2 : 4 : 6-Trinitro-*m*-toluidine may replace picramide. C. HOLLINS.

Manufacture of azo dyes. I. G. FARBENIND. A.-G. (B.P. 268,807, 31.3.27. Ger., 31.3.26).—1-Aminoaryl-5-pyrazolones, prepared from nitroarylhydrazines, are condensed with chloroformic, chloroacetic, or similar esters and the products are coupled with diazo compounds. The resulting azo dyes, if they contain just sufficient sulphonic groups to make them soluble, dye wool-silk mixtures in level shades. Examples are: aniline-*o*-sulphonic acid \rightarrow 1-*p*-urethanophenyl-3-methyl-5-pyrazolone; sulphanilic acid \rightarrow 1-*p*-carbethoxy-methylaminophenyl-3-methyl-5-pyrazolone. C. HOLLINS.

Azo dyes and their application. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, P. CHORLEY, and R. BRIGHTMAN (B.P. 294,922, 28.4.27).—Tetrazotised 2-nitro- or 2 : 2'-dinitrobenzidine is coupled either with 2 mols. of a 2 : 8-aminonaphtholsulphonic acid or with 1 mol. of this and 1 mol. of another coupling component, to give dyes for wool and especially for viscose silk. Examples are: 2-nitrobenzidine with salicylic acid and γ -acid (red), with 1-(2 : 5-dichlorophenyl)-3-methyl-5-pyrazolone and γ -acid (yellow-brown), with naphthionic acid and phenyl- γ -acid (maroon on viscose, copper-brown on wool), or with γ -acid and resorcinol or *m*-phenylenediamine (violet-brown); 2 : 2'-dinitrobenzidine with salicylic acid and γ -acid (yellowish-brown). C. HOLLINS.

Production of direct azo dyes. P. CACCIA (U.S.P. 1,673,311, 12.6.28. Appl., 20.7.26).—An amine coupling component is boiled with sodium cyanide to form the corresponding cyanamido-derivative, which is then coupled with tetrazotised benzidine etc. to give direct cotton colours which leave silk white in unions. 2-Cyanamido-8-naphthol-6-sulphonic acid (from γ -acid) yields a direct blue with tetrazotised toluidine. C. HOLLINS.

Trisazo dye and process of making same. H. JORDAN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,674,114, 19.6.28. Appl., 17.6.26).—Grey-blue to blue direct cotton dyes of the type arylamine (non-hydroxylated) \rightarrow Cleve acid \rightarrow Cleve acid \rightarrow 1 : 8-aminonaphthol- mono- or -di-sulphonic acid (especially H-acid) are described. The arylamines used as first components are Cleve acid, *p*-toluidine, and sulphanilic acid. The dyes are fast to light and discharge to a pure white. C. HOLLINS.

Disubstituted guanidine salts of dyes containing acid groups. R. E. ROSE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,674,128, 19.6.28. Appl.,

2.11.23).—The disubstituted guanidine (especially diphenylguanidine) salts of acid dyes are insoluble in water but soluble in alcohol, ethyl acetate, etc., and are thus useful for colouring nitrocellulose products and spirit varnishes. The salts are precipitated when diphenylguanidine acetate is added to a solution of the dye in water. C. HOLLINS.

Production of dye powders. B. L. WEST, Assr. to NAT. ANILINE & CHEM. CO. INC. (U.S.P. 1,672,920, 12.6.28. Appl., 2.4.26).—A dry dye is intimately mixed with a hydrated salt. C. HOLLINS.

Manufacture of violet vat dyes [of 2-thionaphthen-2'-indoleindigo series. [A] E. FISCHER, H. HEYNA, and C. J. MÜLLER, [B] R. HEIZ and K. THIESS, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,655,692 and 1,655,697, 10.1.28. Appl., [A] 16.2.27, [B] 11.2.27. Ger., [A] 20.2.26, [B] 19.2.26).—See B.P. 266,382; B., 1928, 562.

Manufacture of azo dyes. H. WAGNER and O. SOHST, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,681,602, 21.8.28. Appl., 12.3.25. Ger., 29.3.24).—See B.P. 294,291; B., 1928, 704.

Mordant disazo dyes. W. NEELMEIER and W. REBNER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,660,625, 28.2.28. Appl., 17.3.27. Ger., 24.3.26).—See B.P. 289,135; B., 1928, 518.

Diazo-types (B.P. 294,972).—See XXI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

[Removal from cotton yarns and fabrics of] mineral oil stains. R. F. MENNECKE and L. PICARD (Bull. Soc. Ind. Mulhouse, 1928, 94, 241—243). Reports by V. SILBERMANN, T. BAUMANN, and P. DE CHAMBRIER (*Ibid.*, 243—256, 257, and 258—267).—Complete removal of such stains, arising from the lubricants used in spinning and weaving, by ordinary bleaching processes is not possible, particularly in the case of "aged" stains, unless the oil contains 33—60% of colza oil (cf. Scheurer and Wallach, B., 1911, 277). It is recommended that the addition of easily saponifiable vegetable oils to the lubricants used in spinning and weaving should be compulsory, so that subsequent removal of oil stains may be facilitated. A special oil manufactured by Usines de Pechelbronn, not yet commercially available, was shown to be completely removable even after ageing for 8 months. A. J. HALL.

Effect of heat on cotton. A. H. TILTMAN and B. D. PORRITT (India-Rubber J., 1928, 76, 9—12).—Warp test pieces of a single warp, single weft, scoured balloon cambric, woven from a high-grade Egyptian cotton, were heated in air and *in vacuo* under different conditions of time and temperature, exposed to the air for some hours, and strength tests carried out on a Schopper constant-extension machine. A permanent loss of strength occurs on heating cotton above 120°, and this loss becomes more marked with rise of temperature, until at about 240° almost total destruction occurs in 4 hrs. The effect of heat on the tensile strength is roughly proportional to the time of heating at constant temperature, whilst the loss of strength is greater when cotton is heated in air than *in vacuo* under

the same conditions of time and temperature, this effect being attributed in part to the action of oxygen. Heating does not appear to affect the elastic characteristics of cotton fabric, but only to influence the breaking point.

B. P. RIDGE.

Oxidation of cellulose in the soil. S. WINOGRADSKI (Compt. rend., 1928, 187, 326—330).—In the action of bacterial forms on filter-paper previously described (B., 1927, 294), the loss of weight of the paper under the action of vibrios amounts to 25% after 10—15 days, but does not exceed 50% even after 3—4 weeks. If the paper is dipped in a similar saline solution the loss is less than half of that when it is spread on the surface. The oxidation produced by the vibrios is strictly analogous to that produced by calcium hypochlorite, nitric acid, ammonium persulphate, etc., and in addition to the oxycellulose formed in the fibres, an oxycellulose soluble or dispersible in water is also formed; volatile fatty acids are not produced. During the reaction the alkalinity may rise to p_H 8.5 if the nitrogen is supplied as sodium or potassium nitrate. The mucilage obtained under the action of the cytophaga is completely soluble in 2% soda, but its chemical nature is undetermined, although it is probably an oxidation product, and the transformation, though slower than with the vibrios, is complete. A third group of bacteria attack the paper, slowly at first but ultimately up to 80% decomposition, affording a mucilage which varies from colourless to yellow. The action is very sensitive to the presence of air, and the solubility of the residue may exceed 33%. The resistance of these oxycellulose products to further action of the soil bacteria makes it probable that such products persist in the soil and form part of its organic colloidal content.

R. BRIGHTMAN.

Behaviour of lignin and chlorolignin in the preparation of wood pulp by means of chlorine. H. P. WAENTIG (Z. angew. Chem., 1928, 41, 977—980; cf. B., 1928, 476).—In the de Vains and Pomilio chlorination processes, the preliminary treatment of the material with soda leads to economy in the total consumption of soda. In the pretreatment of wood, high concentration of soda and high cooking pressure both tend to reduce the amounts of soda and chlorine required in the later stages of the process. The net soda consumption is also reduced by utilising the digester-liquor for washing out the chlorolignins. By the extraction of chlorolignin with alcohol it is shown that the rise in temperature of the material during chlorination and the addition of hydrochloric acid increase the yield of extract. Prolonged treatment with chlorine increases the yield of chlorolignin and also its chlorine content. It is suggested that in the process chlorolignin is partially hydrolysed by the water present, and the addition of hydrochloric acid retards this. Further prolonged exposure to chlorine results in the production of more highly chlorinated compounds having greater solubility in alcohol. Hydrochloric acid formed during chlorination causes some degradation of cellulose, but by careful control an α -cellulose content of 95—96% may be attained.

A. G. POLLARD.

Carbohydrate constituents of sodium hydroxide cellulose from pine wood. E. HÄGGLUND and F. W.

KLINGSTEDT (Cellulosechem., 1928, 9, 77—80).—Sodium hydroxide cellulose obtained as a 43% yield from pine wood was found to contain mannan 3.3%, pentosan 5.3%, no galactan, and only a trace of lævulan. After mercerisation of this material with 17.5% sodium hydroxide solution an α -cellulose was obtained which still yielded 2.1% of mannan and 1.5% of pentosan, thus differing from similar material obtained from sulphite-cellulose which contained no hemicellulose. The total mannan content is obtained by first converting the material completely into sugar and then determining the mannose present. Lignin does not interfere with the determination of pentosan in the cellulose.

B. P. RIDGE.

Constitution of cellulose xanthate. T. LIESER (Annalen, 1928, 464, 43—55).—When cellulose xanthate, prepared in the usual manner from purified unbleached cotton yarn, is extracted repeatedly with methyl alcohol until no further colouring matter is removed, a product remains which, after being washed with ether and dried in a vacuum (with periodic admission of dried air etc.), has a very pale greenish-yellow colour, retains the form of the original fibre, and contains 76.6% of cellulose, 14.8% S, and 7.42% Na. It is accordingly concluded that pure cellulose xanthate, obtained now for the first time, has the formula $C_6H_{10}O_5 \cdot C_6H_5O_4 \cdot O \cdot CS \cdot Na$. The high sodium content (5.45% calc.) is not explained, but is possibly due to the presence of sodium hydroxide in additive union. The new formula fits in better with the facts of technical practice than does that of Cross and Bevan, whose formula requires the use of carbon disulphide in amount equal to 47% of the cellulose used, whereas in practice only 30—35% is taken. The new formula requires 23.5% of carbon disulphide, and the excess used in practice is readily explained, since numerous by-products are known to be formed and to be present in ordinary "cellulose xanthate." The new formula further explains why in persulphidised sodium cellulose xanthate not more than 1 mol. of carbon disulphide can be introduced per 2 mols. of cellulose, at any rate under normal conditions of mercerisation. Pure cellulose xanthate is more stable than crude. When dissolved in 7—8% alkali it shows no syneresis on being kept, though the cellulose present passes into cellulose-A, as it does when pure cellulose xanthate is kept in the dry state. This change is due to the combined sodium, for, when the latter is replaced by another metal, the cellulose remains unchanged in type. Cross and Bevan's suggestion, that iodine converts cellulose xanthate, in presence of acetic acid, into a disulphide, is now realised with the pure material. The disulphide is formed quantitatively as a pale yellow, insoluble, stable substance, which retains the fibre-form of its parent substance, and which is reduced by sodium amalgam to cellulose xanthate. Methylated derivatives of cellulose xanthate are not obtainable in anything approaching the pure condition. Methyl sulphate or iodide converts the sodium salt into mixtures (methylation; disulphide formation; hydrolysis). Benzyl chloride behaves similarly. By covering the pure xanthate with neutral solutions of salts of different metals, various metal cellulose xanthates are readily obtained (cadmium, zinc, nickel, cupric, chromic, and

ferrous). The heavy-metal salts are pale yellow, but the colour is masked by the presence of metal hydroxides, formed as the result of the excess of sodium hydroxide already referred to. Attempted formation of the ferric salt leads to disulphide formation, whilst mercuric chloride produces a mercuric salt combined with at least 2 mols. of mercuric chloride. The fibre-like mercuric compound is yellowish, rapidly darkening in the light.

E. E. TURNER.

Viscose. XVI. Spinning. VI. Maturing of alkali-cellulose and its effect on spinning and on the properties of the threads. R. TOMIYASU (J. Cellulose Inst., Tokyo, 1928, 4, 113—116).—Alkali-cellulose which had matured at 12°, 20°, and 25° for varying periods up to 3 days was xanthated and the resulting viscose solutions, after ripening for varying periods, were spun into a bath containing 10 pts. of sulphuric acid, 7.5 pts. of dextrose, 25.54 pts. of crystalline magnesium sulphate, 5.34 pts. of crystalline zinc sulphate, and 52.62 pts. of water at 45°. A study of the physical properties of the filaments thus obtained indicates that (a) the longer the period of maturing of the alkali-cellulose, particularly at the higher temperatures, the longer must the viscose solution be allowed to ripen to make it spinnable, and the thicker are the filaments; (b) at the lower temperatures the time of maturing of the alkali-cellulose has no great influence on the maximum strength of the thread, but at higher temperatures the more highly matured alkali-celluloses give a markedly weaker thread; (c) for the production of the strongest threads the alkali-cellulose should be matured for a moderate time at a medium temperature; (d) a less ripe viscose is required for the production of threads of maximum stretch than is necessary for threads of maximum strength; and (e) of two spinning speeds (23 and 46 m./min.) the higher gave the stronger threads.

D. J. NORMAN.

Viscose. VIII. Effect of dialysis in relation to viscosity. T. MUKOYAMA (J. Cellulose Inst., Tokyo, 1928, 4, 109—112).—Viscose solution (cellulose 4.03%, alkali 3.74%) which had ripened for 24 hrs. was subjected to dialysis using water or varying concentrations (up to 6%) of caustic soda in the dialyser. With water alone the viscose rapidly gelatinised, but with increasing concentrations of caustic soda the viscosity of the viscose solution fell progressively.

D. J. NORMAN.

Influence of wetting on strength and elasticity of artificial silks. Y. KAMI (Kunstseide, 1928, 10, 207).—Cellulose silks suffer a greater loss of strength and elasticity when immersed in boiling water than in cold water, the loss of strength increasing (after a certain minimum period of about 1½ hrs.) with the duration of immersion. The strength and elasticity are further reduced when the wet silk is dried at 98—99°, but not when dried at 27—35°.

A. J. HALL.

Effect of fine division on the copper number of cellulose. K. G. JONAS (Z. angew. Chem., 1928, 41, 960—961).—Soaking in water does not affect the copper number of soda- or sulphite-cellulose, but there is a notable difference between the values for soda-cellulose filings (5.32), teased cellulose (5.39), and beaten

cellulose (5.27) on the one hand, and cellulose cut into pieces 0.6 cm. square (6.35) on the other; the corresponding values for sulphite-cellulose are 2.27, 2.52, 2.25, and 3.40. Beaten cellulose is in the best form for determination of the copper number since it is more easily and rapidly weighed, the nature of the fibre is unchanged, and the cuprous oxide is very readily dissolved out by ferric sulphate and sulphuric acid.

C. HOLLINS.

Primary and secondary cellulose acetates. H. PRINGSHEIM and E. SCHAPIRO (Cellulosechem., 1928, 9, 80—82).—Methods are described for converting the acetone-insoluble, primary cellulose acetate into the acetone-soluble secondary acetate, the change being ascribed to disaggregation of the primary compound. The percentage yield and solubility in acetone of the product obtained by heating the primary acetate with tetralin vary with the time and temperature of heating, the product being a crumbly mass. The secondary acetate is produced by shaking the primary substance in the cold, keeping it several days in the cold, or heating it with benzenesulphonic acid. Changes in acetic acid content, solubility in acetone, viscosity, and film-forming power under different experimental conditions are discussed.

B. P. RIDGE.

Residues of sulphate-turpentine oil. QVIST.—See XX. **Effect of sulphite waste-liquor on river water.** ÖMAN.—See XXIII.

PATENTS.

Continuous manufacture of artificial textile fabrics. LA SOIE D'AUBENTON (F.P. 625,049, 24.11.26).—The precipitated fibres are spun into yarn, passed over rollers, washed, desulphurised, soaped, bleached, and dried on heated drums.

L. A. COLES.

Protection of materials from moth. I. G. FARBER and A. G. (B.P. 274,425, 5.5.27. Ger., 13.7.26).—Wool, skins, hair, feathers, etc. are treated with 2-hydroxy-1-carboxylic acids in which the 5-position is occupied by halogen or sulphur, or in which both 3- and 5-positions are occupied, the 5- by halogen, sulphur, or hydrocarbon groups, the 3- by these or hydroxyl. Examples are: 5-chloro- and 3:5-dichloro-salicylic acids, 5-chloro- and 5-bromo-*o*-cresotic acids, 3:5-dimethylsalicylic acid, 1-chloro-2:3-hydroxynaphthoic acid, and the products (usually 3:3'- or 5:5'-disulphides) are obtained by the action of sulphur monochloride on salicylic acid, *p*-cresotic acid, 5-chlorosalicylic acid, 2:3-hydroxynaphthoic acid, etc.

C. HOLLINS.

Method of pulping wood. F. G. RAWLING (U.S.P. 1,679,682, 7.8.28. Appl., 21.7.26).—Wood chips are treated with sodium sulphite solution and the sodium salt of a weakly ionised polybasic acid at 100—150°. After removing the unabsorbed liquor from the treated wood, the residue is treated with sulphur dioxide at 100—160°.

H. ROYAL-DAWSON.

Manufacture of artificial silk. H. P. BASSETT (U.S.P. 1,676,003, 3.7.28. Appl., 15.12.25).—A cuprammonium solution of cellulose is spun into caustic alkali solution of *d* 1.381 and is washed with alkali of *d* 1.115—1.16 and then with alkali solution of less strength.

D. J. NORMAN.

Manufacture of nitrocellulose. I. G. FARBENIND. A.-G. (B.P. 280,922, 16.11.27. Ger., 22.11.26).—Nitrocellulose of suitably low viscosity is obtained by nitrating cellulose that has been heated with water under pressure at 140–170° for a prolonged time.

S. S. WOOLF.

Continuous manufacture of cellulose acetate. SOC. CHIM. DES USINES DU RHÔNE (B.P. 274,814, 6.4.27. Fr., 26.7.26).—All the necessary operations are carried out in one or more inclined, tubular, reaction vessels rotating about their axes and preferably provided internally with mixing and circulating devices, the particular type of device being determined by the texture of the reaction mixture at the point at which it is to operate. The various reagents may be introduced at the end of the reaction vessel, or at any point along the path followed by the reacting mass. The temperature may be controlled by means of sprays.

D. J. NORMAN.

Productions of threads, bands, etc. of cellulose esters and ethers. PATHÉ CINÉMA (ANC. ÉTABL. PATHÉ FRÈRES) (F.P. 624,278, 3.3.26).—Solutions of the dry ester in a dry, non-aqueous solvent, to which plasticising agents may be added, are coagulated by passing into water.

L. A. COLES.

Production of colour compositions and their application in the printing industry. Manufacture of "carbon papers" and typewriter ribbons. R. D. MAIN, E. G. NIXON, and LAMSON-PARAGON SUPPLY CO., LTD. (B.P. 295,118—9, 18.7.27).—(A) Non-smearing colour compositions for transfer or "carbon" papers in which the oils, fats, and waxes commonly used for this purpose are replaced wholly or mainly by synthetic products, viz., soluble "resoles" of the phenol-aldehyde, urea-aldehyde, or coumarone resin types, or cellulose esters. An organic solvent may be added. Examples are: (1) lamp-black, china clay, cellulose nitrate, acetone, and toluene; (2) a purple lake, china clay, phenol-formaldehyde "resole," cyclohexanol, acetone, and methylated spirit. (b) The pastes are applied to the carrier sheets in the cold.

C. HOLLINS.

Transforming the fibres of *Corchorus capsularis* and such like products and derivatives into a product resembling wool. P. CELLI (B.P. 290,071, 21.7.27).—See F.P. 613,973; B., 1928, 187.

Manufacture of wool-like cellulosic material. W. HARRISON (U.S.P. 1,680,021, 7.8.28. Appl., 26.1.27. U.K., 12.2.26).—See B.P. 268,505; B., 1927, 473.

Manufacture of cellulose derivatives. L. LILIENFELD (U.S.P. 1,682,292 and 1,682,294, 28.8.28. Appl., 23.7.24. Austr., 4.4.24).—See B.P. 231,809 and 231,810; B., 1925, 914.

Manufacture of artificial materials [from cellulose derivatives]. L. LILIENFELD (U.S.P. 1,682,293, 28.8.28. Appl., 23.7.24. Austr., 16.7.28).—See B.P. 231,811; B., 1925, 914.

Manufacture of threads, films, etc. from cellulose derivatives. E. BERL (U.S.P. 1,679,850, 7.8.28. Appl., 11.3.25. Ger., 17.3.24).—See B.P. 230,813; B., 1925, 877.

Treatment and utilisation of black liquor obtained in manufacture of soda and sulphate cellulose. E. HÄGGLUND (U.S.P. 1,680,540, 14.8.28. Appl., 18.6.27. Swed., 21.6.26).—See B.P. 273,267; B., 1928, 188.

Production of carbohydrate compounds. W. HARRISON (U.S.P. 1,680,020, 7.8.28. Appl., 4.10.26. U.K., 24.10.25).—See B.P. 264,261; B., 1927, 552.

Esterification of cellulose in presence of a phenol. C. DIAMOND, ASSR. to COURTAULDS, LTD. (U.S.P. 1,681,103, 14.8.28. Appl., 14.4.27. U.K., 21.6.26).—See B.P. 269,012; B., 1927, 473.

Manufacture of pulp for paper production. D. R. NANJI (U.S.P. 1,679,441, 7.8.28. Appl., 5.11.26. U.K., 17.8.26).—See B.P. 280,629; B., 1928, 155.

Manufacture of [matt] cellulose ester or the like sheets. NON-INFLAMMABLE FILM CO., LTD., and H. J. MALLABAR (B.P. 295,797, 27.6.27).

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Treatment [dyeing] of union materials containing cellulose acetate silk. K. WOLFGANG (Kunstseide, 1928, 10, 117—118).—In dyeing wool and cellulose acetate silk materials at high temperatures with acid dyes, 15 g. of magnesium sulphate are added per litre of dye liquor in order to preserve the lustre of the cellulose acetate silk (cf. B.P. 246,879; B., 1926, 317). A soap liquor suitable for degumming real silk present in cellulose acetate silk materials consists of 10 g. of Marseilles soap and 3 g. of sodium phenoxide, the last-named substance having no hydrolysing action on the cellulose acetate silk.

A. J. HALL.

Absorption of dyes by mineral salts. MICHELS.—See XIII.

PATENTS.

Bleaching of fabrics. ROHM & HAAS CO., ASSEES. of C. S. HOLLANDER (B.P. 266,691, 8.2.27. U.S., 25.2.26).—"Marking-off" in kier-boiling is prevented by adding to the alkaline liquor a mono- or di-cyclic substance possessing or developing a quinonoid structure. Examples are: *p*-nitrosophenol, nitroso-*m*-cresol, *p*-nitrosodimethylaniline.

C. HOLLINS.

Dyeing of cellulose esters or ethers. I. G. FARBENIND. A.-G. (B.P. 272,482, 26.5.27. Ger., 8.6.26).—Cellulose acetate silk is dyed with ethers of 1:4-diamino-2-hydroxyanthraquinone. The methyl and ethyl ethers give magenta shades. [Stat. ref.]

C. HOLLINS.

Production of yellow dyeings on cellulose esters or ethers. E. FISCHER and C. E. MÜLLER, ASSRS. to GRASELLI DYESTUFF CORP. (U.S.P. 1,674,168, 19.6.28. Appl., 19.7.27. Ger., 29.7.26).—Acetate silk is dyed in fast, non-phototropic shades with azo dyes obtained by condensing 4-aminoazobenzene with 1-chloro-2:6-dinitrobenzene-4-sulphonic acid or with 1-chloro-4-nitrobenzene-2-sulphonic acid.

C. HOLLINS.

Mordant dyeing of materials made with or containing cellulose esters and ethers. BRIT. CELANESE, LTD., G. RIVAT, and E. CADGÈNE (B.P. 273,692, 22.6.27. U.S., 30.6.26).—The material is mordanted with a

40–55% solution of a ferric salt at 40–60°, and can then be dyed with logwood or other vegetable dyes.

C. HOLLINS.

Treatment of yarn packages with liquids and products obtained thereby. [Effect dyeing etc. of cellulose ester or ether materials in the form of tightly-wound yarn.] BRIT. CELANESE, LTD. (B.P. 269,195, 8.4.27. U.S., 12.4.26).—Effect dyeings are produced by immersing cops, spools, bobbins, etc. of cellulose ester or ether silk yarn in an organic liquid containing dye or stripping agent and removing them before the colour change has taken place throughout the winding. Multiple colour effects result when several dyes of different penetrating power are present in the liquid, or when successive immersions are made in different dye solutions.

C. HOLLINS.

Producing [pattern] effects on pile fabrics. H. LIVSEY, G. E. HOLDEN, and J. & J. M. WORRALL, LTD. (B.P. 295,203, 15.3.28).—Pattern effects are obtained by printing pile fabric with an oxidising agent, and then singeing to burn away the pile in the printed parts.

A. J. HALL.

Treatment [mercerisation] of materials containing artificial [regenerated cellulose] silk. BLEACHERS' ASSOC., LTD., W. KERSHAW, F. L. BARRETT, C. J. WHITELEGG, and G. D. SUTTON (B.P. 295,062, 9.5.27).—Cotton materials containing regenerated cellulose silk, such as cuprammonium and viscose silks, are mercerised with caustic soda of about d 1.265 under the usual conditions except that the washing of the material free from alkali is effected with a comparatively large volume of water (at not less than 50°, preferably 100°); the deleterious action of the alkali during the washing process is thus almost completely avoided.

A. J. HALL.

[Mercerisation] processes in which viscose silk is subjected to the action of caustic alkalis. SILVER SPRINGS BLEACHING & DYEING CO., LTD., and A. J. HALL (B.P. 295,488, 9.7.27).—Cotton materials containing desulphurised viscose silk are satisfactorily mercerised or similarly treated by means of caustic potash instead of caustic soda, the lustre, strength, and softness of handle of the viscose silk being inappreciably affected. It is preferred to use the mercerising liquors at above 15°, since their effect on the silk diminishes with rise of temperature: caustic potash of d 1.35 exerts the greatest deleterious action on viscose silk. *E.g.*, cotton fabric (cotton warp and viscose silk weft) is mercerised so that the cotton obtains its maximum increase of lustre by impregnation with caustic potash of d 1.30 at 30°, stretched, washed with water, soured, washed, and dried; the whole process occupies about 80 sec.

A. J. HALL.

Improving [surfacing] products [fabrics] obtained from vegetable fibres. M. MELLAND, Assee. of O. DUBAC (B.P. 273,327, 24.6.27. Ger., 25.6.26).—Special wool-, silk-, or linen-like properties, dependent on the original characteristics of the fabric, are conferred on cotton fabrics by subjecting them successively to the usual process of mercerisation with cold caustic alkali and to the process of B.P. 254,695 (B., 1927, 247), now termed "melianising," which denotes a brief treatment with hot or boiling concentrated caustic alkalis. Loosely

woven cotton fabric, after being "melianised" and thereby gaining a wool-like handle but no increased lustre, becomes silk-like when mercerised afterwards, whereas a closely woven fabric becomes more flexible but not wool-like after melianising, and linen-like when mercerised afterwards. A linen-like fabric is produced by treating a coarse cotton fabric (not singed or desized) for 5 min. with a 60% solution of caustic soda at 88°, then washing, and mercerising with caustic soda of d 1.19.

A. J. HALL.

Dyeing of furs, hairs, feathers, etc. 1. G. FARBERIND. A.-G., Assee. of O. KALTWASSER and H. KIRCHHOFF (U.S.P. 1,643,246, 20.9.27. Appl., 9.8.26. Ger., 10.7.25).—See F.P. 618,723; B., 1928, 86.

Treatment of cellulose derivatives. G. H. ELLIS, H. C. OLPIN, W. B. MILLER, and CELANESE CORP. OF AMERICA (U.S.P. 1,679,935, 7.8.28. Appl., 15.8.27. U.K., 30.9.26).—See B.P. 282,253; B., 1928, 189.

Azo dyes (B.P. 291,922).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Pump for molten salts [especially caustic alkalis]. H. OETTINGER (Chem. Fabr., 1928, 516–518).—The pump is designed for the filling of caustic soda or potash in the molten state from the pots into drums. A plunger pump is driven by a long vertical shaft from a vertical motor at the top, the whole being portable. It is of the glandless type, and is lubricated by the alkali itself. All parts in contact with the latter are of a special alkali-resistant cast alloy. It will empty a 12-ton pot in 2 hrs., requires $1\frac{1}{2}$ h.p., and makes 950 r.p.m. The construction of the suction device varies according to whether the settlings or the clear caustic are to be pumped. The wear on the pump is not excessive. A method of filling a number of drums without stopping the pump is described. C. IRWIN.

Analysis of nitrates. P. RISCHBIETH (Chem.-Ztg., 1928, 52, 691).—Pure iron is dissolved in hydrochloric acid in a flask with a short, wide neck, through which carbon dioxide is passed. The outlet tube is connected to either a beaker of water or a gas pipette. The nitrate is added by removing the stopper while the gas continues to pass. After any air has been removed, the tube is connected to the gas pipette containing strong caustic potash, and the flask warmed. The nitric oxide evolved is measured and the residual ferrous iron titrated, the two results affording a check on each other.

C. IRWIN.

Preparation of cyanides from calcium cyanamide. A. A. YAKOVKIN and N. A. FLEISHER (Trans. State Inst. Appl. Chem., Moscow, 1927, No. 5, 3–7).—On calcination at 900–1000° in a current of nitrogen with sodium carbonate, calcium cyanamide containing 12.7% N, 1.4% of gypsum, and no calcium chloride yielded very little cyanide, most of the nitrogen volatilising as ammonia. With freshly-prepared calcium cyanamide containing 20.6–22.2% N, the yield of cyanide at 950° (optimum) for 2 hrs. was increased by the presence of small quantities of cerium, magnesium, or aluminium nitrides. The best yield (89%) was

obtained with 1.62 g. of potassium chloride for 0.5 g. of calcium cyanamide, and 0.05 g. of magnesium nitride.

CHEMICAL ABSTRACTS.

Basic lead acetate. E. SOMMER (Z. Zuckerind. Czechoslov., 1928, 52, 644—651).—A series of solutions of basic lead acetate was prepared, all in the usual manner, but using different grades of commercial litharge. The most striking result was obtained with a grade of flake litharge, a solution containing 34.2% PbO (basic), with only 7.2% of residue resulting. In the other solutions the content in basic PbO varied from 15.0 to 34.2%. Using 1 pt. of the normal acetate and 2 pts. of litharge, a solution containing as much as 66.66% PbO (basic) was prepared, but it was not possible further to increase the basicity by using more litharge. From "superbasic" solutions thus made it was possible to precipitate the dibasic lead compound quantitatively by the addition of alcohol (9:1), ordinary basic lead acetate remaining in solution. J. P. OGILVIE.

PATENTS.

Apparatus for the [continuous] formation of nitric acid or hydrochloric acid. G. GUADAGNI (U.S.P. 1,680,019, 7.8.28. Appl., 13.1.25. Ger., 12.12.24).—A single elongated container, with a fire-proof cover, is mounted completely in an oven so that burnt gases circulate all about the container. The reagents are fed in regulated quantities through an inlet at one end of the cover projecting through the oven roof, and a delivery tower at the other end discharges gaseous products. A tube for the continuous discharge of bisulphate has its inlet end dipping in the liquid near the gaseous discharge and remote from the chemical inlet, whilst its outlet passes through the container at the normal liquid level. W. G. CAREY.

Concentration of dilute nitric acid. NORSK HYDRO-ELEKTRISK KVAELSTOFAKTIESELSKAB (B.P. 281,642, 23.11.27. Norw., 6.12.26).—Dilute nitric acid is subjected to continuous preliminary concentration by boiling under reduced pressure and rectifying. The process is carried out in several stages, and acid vapour from the stronger nitric acid is led into a vessel placed before the others containing boiling weaker acid. W. G. CAREY.

Scrubbing waste acid gases [from the manufacture of nitric acid]. BAMAG-MEGUIN A.-G. (F.P. 624,847, 19.11.26).—The gases are scrubbed with milk of lime in towers. L. A. COLES.

Manufacture of sulphuric acid. SOC. GÉN. MÉTALL. DE HOBOKEN (B.P. 285,847, 15.12.27. Ger., 23.2.27).—A solution ("nitrose") of nitrous products in sulphuric acid ($d > 1.59$) is injected in atomised form, either above or below the surface of the liquid in lead chambers. The strength of the injected acid is higher than that of the acid on the floor, and the nitrous solution is injected in such quantity that the acid on the floor still contains nitrous products. The acid from the last chamber is injected into the atmosphere of the preceding chamber, and so on, the circulating system comprising a chamber regulator and, if necessary, a denitrating tower, or all the Gay Lussac apparatus and the first Glover tower. W. G. CAREY.

Manufacture of sulphuric acid. METALLBANK & METALLURGISCHE GES. A.-G. (F.P. 624,913, 20.11.26. Ger., 10.7. and 6.10.26).—Gases from the pyrites burners are freed from arsenic and selenium in electrical gas purifiers, the acid mist is precipitated as sulphuric acid, and residual gases pass into the contact chambers. L. A. COLES.

Utilisation of gas mixtures containing varying proportions of sulphur dioxide and water vapour for the manufacture of sulphuric acid etc. METALLBANK & METALLURGISCHE GES. A.-G. (F.P. 624,912, 20.11.26. Ger., 25.11.25).—A gas mixture containing a high proportion of sulphur dioxide is used for the manufacture of sulphuric acid or sulphite liquor, and gas containing a low proportion is added at a point where the additional water vapour etc. exerts no harmful influence. L. A. COLES.

Production of hydrocyanic acid. A. R. FRANK and N. CARO (B.P. 282,379, 5.12.27. Ger., 17.12.26).—A mixture of ammonia and carbon monoxide or gases containing these are passed at ordinary or increased pressure over cyanamides of the alkaline earths and/or of magnesium at 700—800°, or over compounds, e.g., oxides, carbonates, etc., which can be converted into cyanamides with hydrocyanic acid or the ammonia-carbon monoxide mixture. The process is carried out in the absence of activated carbon and of metals causing decomposition, or this decomposing action is prevented by the addition of "poisoning" substances (e.g., sulphides) or water. W. G. CAREY.

Concentration of acetic acid solution. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 295,238, 4.5.27).—The acetic acid is extracted with an organic base not attacked by concentrated acetic acid, e.g., quinoline, quinaldine, dimethyl- or diethyl-aniline, the concentrated acid then being recovered by distillation. A solvent for the acetic acid, substantially insoluble in water, may be added, e.g., benzene, chloroform, or trichloroethylene. W. G. CAREY.

Temperature control in synthetic production of ammonia and the like. H. SVANOE, ASST. to AMMONIA CORP. (U.S.P. 1,678,964, 31.7.28. Appl., 3.4.26).—The initial reaction is modified, and the conduction of heat improved, by introducing into the catalytic chamber a gas mixture containing excess of hydrogen. A mixture containing excess of nitrogen is subsequently used. F. G. CLARKE.

Manufacture of alkali hydroxides. L. P. CURTIN (U.S.P. 1,678,767, 31.7.28. Appl., 24.4.26. Renewed 17.1.28).—Alkali sulphide is slowly added to an agitated aqueous suspension of lead monoxide, accumulation of sulphide ions being avoided. F. G. CLARKE.

Soda ash furnace. D. R. MEANS, ASST. to PITTSBURGH PLATE GLASS CO. (U.S.P. 1,679,486, 7.8.28. Appl., 14.10.26).—A covered calcining pan for the soda is fitted with agitators and discharges calcined material at one end into a rotary furnace, heated gases from a furnace beneath the pan being conducted along the shell of the rotary furnace. W. G. CAREY.

Apparatus for crystallising salts. KALI-IND. A.-G. (G.P. 448,189, 4.12.24).—Hollow devices having a perfectly smooth surface and not provided with scrapers

rotate with a circumferential velocity of at least 6 m./sec., below the level of the solution. L. A. COLES.

Separation of alkali salts. E. WEITZ (B.P. 271,869, 20.5.27).—Separation of sodium or potassium salts of different acids or of sodium and potassium salts of the same or of different acids is facilitated by modifying the solubility of the salts by saturating solutions of them with ammonia; *e.g.*, after crystallising and removing potassium nitrate from solutions of sodium nitrate and potassium chloride, further quantities of potassium nitrate are thrown out of solution by saturation with ammonia. Alternatively, the dry mixture, *e.g.*, the product obtained by "azotising" a mixture of carbon, sodium carbonate, and iron, may be leached with concentrated ammonia, in this case only the sodium cyanide passing into solution. L. A. COLES.

Production of calcined alkali dichromates in a powdered form or compressed into tabloids etc. V. ZIEREN (G.P. 448,799, 31.12.25).—Sufficient previously calcined dichromate is added to dichromate solution flowing into a calcining apparatus to yield a loose product, and this is cooled with constant stirring or compressed. L. A. COLES.

Precipitation of barium chloride from solution. RHEINANIA-KUNHEIM VER. CHEM. FABR. A.-G., Assees. of F. RÜSBERG and E. FÖHRENBACH (G.P. 448,199, 10.10.24).—Gases containing hydrogen chloride are passed over active charcoal etc. and/or through concentrated hydrochloric acid containing a small quantity of barium chloride in suspension, preferably after treatment with concentrated sulphuric acid, and are then passed into the barium chloride solution. L. A. COLES.

Production of solid calcium nitrate. I. G. FARBENIND. A.-G. (Swiss P. 119,726, 26.10.25. Ger., 27.10.24 and 13.2.25).—Small quantities of ammonium compounds or salts, or compounds capable of yielding ammonia, *e.g.*, urea, are added to concentrated calcium nitrate solutions before drying, *e.g.*, on revolving cylinders or by atomisation. L. A. COLES.

Production of cyanamide from calcium cyanamide. H. C. HETHERINGTON and L. A. PINCK, Assrs. to A. B. LAMB (U.S.P. 1,673,820, 19.6.28. Appl., 2.3.26).—Crude calcium cyanamide is mixed with an equal weight of water and at once filtered, the filter-cake is washed with water at 60–75°, the washings being cooled to 20°, and the filtrate and washings are treated as usual with dilute acid. The cyanamide produced is practically free from dicyanodiamide. C. HOLLINS.

Production of nitrogen compounds etc. W. SCHWARZENAUER (F.P. 622,266, 29.9.26. Ger., 3.11.25, 12.4., 5.9., and 8.9.26).—The by-products and residual energy in the production of oxides of nitrogen and their conversion into calcium nitrate, and in the synthesis of ammonia by the cyanide process, are used for the decomposition of phosphates, for working internal-combustion engines, etc., and the synthetic ammonia and the carbon dioxide in the combustion gases from the engines are used to convert calcium sulphate into calcium carbonate and ammonium sulphate. L. A. COLES.

Manufacture of dicalcium [calcium hydrogen] phosphate and sulphate of ammonia from phos-

phoric acid or its acid salts, calcium sulphate, and ammonia. R. HADDAN. From E. R. STACKABLE (B.P. 293,942, 19.5.27).—Phosphoric acid (preferably commercial 80%), calcium sulphate (preferably the dihydrate), and ammonia react to form calcium hydrogen phosphate and ammonium sulphate, thus: $\text{H}_3\text{PO}_4 + \text{CaSO}_4 + 2\text{NH}_3 = (\text{NH}_4)_2\text{SO}_4 + \text{CaHPO}_4$, the components being used in the stoichiometrical proportions. The phosphoric acid may be replaced by a suitable proportion of commercial acid calcium phosphate (calcium dihydrogen phosphate mixed with calcium sulphate) or especially of the similar product obtained by treating phosphate rock or bone phosphate with sulphuric and phosphoric acids so that the ratio $\text{CaSO}_4 : \text{P}_2\text{O}_5$ (water-soluble) = 1 : 1. The ammonia may be gaseous, dissolved, or liquid, but preferably there is used the moist gas diluted with an inert gas to remove the heat of reaction. Alternatively, the phosphoric acid is converted into diammonium hydrogen phosphate, which is then treated with calcium sulphate. B. FULLMAN.

Decomposition of insoluble phosphates. H. HENGSTMAN (F.P. 621,220, 10.9.26. Ger., 10.9.25).—Apatite, phosphorite, etc. are rendered citrate-soluble by passage in a finely-divided form through an electric arc. L. A. COLES.

Production of calcined phosphates. RHEINANIA-KUNHEIM VER. CHEM. FABR. A.-G., Assees. of F. ROTHE and H. BRENEK (G.P. 447,665, 5.11.25).—The citrate-solubility of the product is improved by calcining phosphorites etc. with a suitable flux by means of combustion gases from which constituents combustible with difficulty have been removed by passage through a special combustion chamber provided with means for removing mineral residues from the gases. L. A. COLES.

Treatment of zinc sulphate [to form pigments]. SOC. GÉN. MÉTALL. DE HOBOKEN (B.P. 282,706, 27.5.27. Fr., 30.12.26).—Zinc sulphate and raw blende, and/or raw sulphides, roasted sulphides, or raw sulphides and carbonaceous material, are caused to react in a sintering apparatus working with blown or drawn blast, in such quantities that the sulphate is completely reduced to oxide in sintered form suitable for subsequent reduction. Slags from lead and copper blast furnaces may be added to facilitate the sintering. W. G. CAREY.

Production of aluminium oxide and other aluminium compounds. T. P. HAGLUND (B.P. 295,227, 2.5.27).—Aluminium ores containing sulphur compounds are treated, *e.g.*, with water or steam, so that aluminium sulphide is converted into aluminium oxide, while iron, titanium, and calcium sulphides etc. remain unattacked, and the product, after calcination if necessary, is treated with chlorine or its compounds to convert the sulphides into chlorides, which are then removed, *e.g.*, by sublimation or leaching. L. A. COLES.

Production of aluminium and other metal salts free from iron. I. G. FARBENIND. A.-G., Assees. of C. MÜLLER and L. SCHLECHT (G.P. 448,848, 13.9.24. Addn. to G.P. 410,927; B., 1925, 546).—The process described previously for chromium salts is used. L. A. COLES.

Production of lead tetraethyl. H. W. DAUDT (B.P. 279,106, 14.10.27. U.S., 15.10.26).—Magnesium ethyl chloride is made to react with lead salts. *E.g.*, 30 pts. of ethyl chloride are added to 10 pts. of magnesium turnings, and the mixture is suspended in 125 pts. of ether containing 1 pt. of methyl iodide and a crystal of iodine. The solution is added to a suspension of 60 pts. of lead chloride in 125 pts. of ether maintained meanwhile and for a further 5 hrs. at 0–50°.

B. FULLMAN.

Radioactive substances and preparations thereof.

R. FISCHER (B.P. 257,957, 6.9.26. Switz., 24.7.26).—A solvent for radioactive emanations is prepared by adding an acid containing an amino-group, *e.g.*, 0.1% of diaminohexoic acid hydrochloride, to a suspension of an organic colloid, *e.g.*, gelatin, in an aqueous solution of an alkali or alkaline-earth halide, *e.g.*, physiological salt solution, and then adding an alkali or alkaline-earth hydroxide until the solution has pH 7.4–8.0.

L. A. COLES.

Catalysts of high mechanical strength.

I. G. FARBENIND. A.-G. (B.P. 286,284, 14.10.27. Ger., 3.3.27).—One component of the catalyst, *e.g.*, zinc oxide, is made into a solid mass with one or more water-soluble magnesium salts, and, after heating to decompose the magnesium salt or salts, the product is impregnated with solutions of the other components of the catalyst, *e.g.*, chromic acid.

W. G. CAREY.

Treatment of silicious materials [for the recovery of silica gel and titania]. P. SPENCE & SONS, LTD., T. J. I. CRAIG, and A. KIRKHAM (B.P. 291,681, 28.4.27).—The silicious residue obtained by the acid extraction of some clays or bauxites is digested under pressure with a solution of sodium carbonate such that there is at least 1 mol. of sodium oxide to 1 mol. of silica. The resulting sodium silicate solution is decomposed by addition of sodium hydrogen carbonate or by treatment with carbon dioxide, whilst the concentrated titania residue is worked for the recovery of titania by acid treatment.

A. R. POWELL.

Production and treatment of hydrogen peroxide and other liquids readily giving off active oxygen. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 295,137, 16.2.27).—Cathodes and other metallic apparatus used in the preparation and treatment of hydrogen peroxide etc. consist of or are coated with highly polished silver or base metals or their alloys, *e.g.*, V2A steel, free from mercury. Salts or acids which do not attack the metals are used for rendering the solutions electrically conductive.

L. A. COLES.

Production of hydrogen. R. WILLIAMS, ASSR. to LAZOTE INC. (U.S.P. 1,673,032, 12.6.28. Appl., 5.8.26. Renewed 8.11.27).—In the production of hydrogen from hydrocarbons and steam passed over a catalyst (nickel chromate) at about 500°, unsaturated hydrocarbons are reduced either by a prior catalytic treatment with hydrogen or by adding hydrogen to the mixture and allowing both reactions to proceed together.

C. HOLLINS.

Production of hydrogen by decomposing saturated or unsaturated hydrocarbons or gaseous mixtures containing the same. R. BATTIG (B.P.

271,483, 18.5.27. Ger., 20.5.26).—Methane etc. is passed successively through a preheated chamber containing a lattice-work of bricks, apparatus heated to the temperature necessary for decomposition, a chamber in which carbon is deposited, and a cooling chamber also packed with bricks. When the last chamber is heated sufficiently, the supply of gas is shut off and oxygen or gas containing it is passed through the apparatus in the reverse direction to burn away the carbon and to re-heat the preheating chamber.

L. A. COLES.

Recovering combined nitrogen from crude calcium cyanamide. G. H. BUCHANAN and P. W. GRIFFITH, ASSRS. to AMER. CYANAMID CO. (U.S.P. 1,678,721, 31.7.28. Appl., 29.2.24).—“Lime-nitrogen” is mixed with 1–4 times its weight of water, and after filtration the separated solids are treated in an autoclave with additional water.

F. G. CLARKE.

Production of phosphoric acid. H. LANG, ASSR. to I. G. FARBENIND. A.-G. (U.S.P. 1,680,625, 14.8.28. Appl., 20.10.25. Ger., 23.10.24).—See B.P. 241,903; B., 1926, 273.

Purification of gases [for synthesis of ammonia]. F. JOST, ASSR. to K. MÜLLER (U.S.P. 1,681,702, 21.8.28. Appl., 8.11.26. Ger., 12.11.25).—See B.P. 261,388; B., 1927, 555.

Removal of ammonia from synthesis gases. R. E. SLADE and K. GORDON, ASSRS. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,679,792, 7.8.28. Appl., 28.1.26. U.K., 30.1.25).—See B.P. 241,071; B., 1925, 957.

Production of pure alumina and alkali carbonates. F. ROTHE and H. BRENEK, ASSRS. to RHENANIA VER. CHEM. FABR. A.-G. (U.S.P. 1,680,066, 7.8.28. Appl., 6.10.25. Ger., 13.10.24).—See B.P. 241,232; B., 1926, 708.

Manufacture of aluminium sulphate. R. M. MEIKLEJOHN, ASSR. to GEN. CHEM. CO. (U.S.P. 1,679,366, 7.8.28. Appl., 6.1.26. Can., 2.10.25).—See Can. P. 263,596; B., 1927, 778.

Manufacture of gels of tungstic, stannic, aluminium, and titanium oxides. W. A. PATRICK, ASSR. to SILICA GEL CORP. (U.S.P. 1,682,239—1,682,242, 28.8.28. Appl., [A] 18.2.21, [B] 12.5.24, [C, D] 24.5.24).—See B.P. 212,065; B., 1924, 379.

Production of zirconium compounds. F. RÜSBERG and P. SCHMID, ASSRS. to RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G. (U.S.P. 1,681,195, 21.8.28. Appl., 12.11.27. Ger., 13.12.26).—See B.P. 287,424; B., 1928, 366.

Catalyst compact [for production of hydrogen]. W. SCHULTZE, ASSR. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,680,807, 14.8.28. Appl., 16.8.23).—See B.P. 220,649; B., 1926, 13.

Manufacture of pure sulphur. W. H. BENTLEY, ASSR. to J. RILEY & SONS, LTD. (U.S.P. 1,680,079, 7.8.28. Appl., 16.8.26. U.K., 17.8.25).—See B.P. 252,938; B., 1926, 666.

Acid-proof articles (G.P. 448,606).—See VIII. **Mixed fertilisers** (B.P. 292,068, 295,477, and 295,825).—See XVI.

VIII.—GLASS; CERAMICS.

Plasticity. V. Plastic masses of various inorganic substances and their ceramic utilisation.

O. RUFF and A. RIEBETH (*Z. anorg. Chem.*, 1928, 173, 373—394; cf. B., 1924, 748).—All solids which are wetted by a suitable liquid which does not dissolve them will form a plastic mass of the second order with that liquid if they are in a sufficiently finely-divided form. Silica, tungsten trioxide, and red phosphorus give good plastic masses with water alone; other solids require an acid electrolyte and the strength usually increases with decrease of p_H . The formation of these masses is due to electrolytic dissociation of an adsorption complex with the generation of an electric charge on the surface of the solid and the formation of liquid shells around the solid particles. Alumina when stirred in 0.05—0.1*N*-hydrochloric acid forms a slip which, when cast in plaster moulds, air-dried, and burnt at 900°, then at 1600°, yields dense, hard, and strong ware. For making zirconia crucibles the pure oxide is fired at 1200° and ground for 20 hrs. in water, hydrochloric acid is stirred in to 0.2*N*, the supernatant liquor removed after 2 days' settling, and the plastic mass dried at 120° to 2.5% moisture and pressed into shape under 1000 atm. The shapes after burning at 2200° form a fine porcelain-like mass which will not fracture when quenched from a white heat. Good results are obtained from a zirconia slip made from oxide which has been calcined at 1400° and ground in 0.2*N*-hydrochloric acid; the slip is cast in plaster, and the shapes are slowly dried, preheated at 1000°, brought slowly up to 1200—1500°, and finally fired at 1700°. Ceria and titania crucibles are made similarly, using *N*-hydrochloric acid and burning temperatures of 1500° and 1400°, respectively. Carborundum gives good results when ground with *N*-hydrochloric acid and the castings are fired at 1500°, but magnesia shapes obtained by any of the above procedures usually break on firing.

A. R. POWELL.

PATENTS.

Optical glass. R. J. MONTGOMERY, ASSR. TO BAUSCH & LOMB OPTICAL CO. (U.S.P. 1,681,704, 21.8.28. Appl., 16.4.23).—The ingredients of the batch consist of silica 69.5, sodium oxide 15.8, boric oxide 0.3, commercial didymium oxide 12.0, antimony oxide 1.9, and arsenic oxide 0.5%.

F. G. CROSSE.

Colour composition for vitreous enamel. H. V. HUBER and T. M. FELTON, ASSRS. TO VITRO MANUF. CO. (U.S.P. 1,673,679, 12.6.28. Appl., 2.7.26).—An enamel for glass which does not blacken on firing comprises a mixture of 16.7% of cadmium sulphide, 1.7% of selenium, 76% of lead borate, and 5.6% of cadmium oxide. The two last-named components are fused together to form a clear flux which is then ground with the "cadmium red."

A. R. POWELL.

Treatment of [massive] silica articles. E. HERZOG, ASSR. TO GEN. ELECTRIC CO. (U.S.P. 1,680,681, 14.8.28. Appl., 21.1.26).—The articles are heated to an ambient temperature of about 1000°, the surface is fused locally, and the product annealed before cooling occurs.

H. ROYAL-DAWSON.

Circular kiln [for ceramic products]. T. C. and W. O. PROUTY, ASSRS. to AMER. ENCAUSTIC TILING CO., LTD. (U.S.P. 1,674,992, 26.6.28. Appl., 7.3.25. Renewed 16.5.28).—A circular muffle kiln is provided with means for moving material through it and with independently controlled sources of heat which are applied to separate successive sections of the walls.

A. R. POWELL.

Manufacture of refractory articles. DEUTS. GOLD- U. SILBER-SCHNEIDANSTALT, VORM. ROESSLER (B.P. 292,958, 26.6.28. Ger., 27.6.27).—Magnesia or magnesium chloride containing magnesia, produced from magnesium chloride by treatment with air or steam, is moulded, using water or other suitable liquid, and fired. Zirconium chloride may be added to increase the strength and density of the product.

W. G. CAREY.

Fireproof and acid-proof articles. G. KALLEN (G.P. 448,606, 5.11.24).—Metals oxidisable at high temperatures, e.g., iron, nickel, aluminium, or copper, are coated with material containing zirconium, and heated strongly. The metals partially oxidise and combine with the layer of material, forming a highly resistant coating.

L. A. COLES.

[Self-dressing] abrasive articles. CARBORUNDUM CO., LTD., ASSEES. OF M. L. HARTMANN (B.P. 273,679, 10.6.27. U.S., 3.7.26).—The articles comprise uniform grains of abrasive material and of friable material sufficiently hard to break out of the mass without "glazing" the surface, e.g., calcined clay grog, and binding material, e.g., clay, feldspar, synthetic resins, glue, etc.

L. A. COLES.

Manufacture of refractory heat-insulating material. B. T. HORSFIELD, ASSR. TO ALUMINUM CO. OF AMERICA (U.S.P. 1,682,675, 28.8.28. Appl., 7.12.25).—See B.P. 262,403; B., 1927, 300.

Apparatus for drawing sheet glass. H. HERMANN (B.P. 271,444, 5.5.27. Czecho-Slov., 20.5.26).

IX.—BUILDING MATERIALS.

Heat balance in rotary cement kilns. H. POOLEY, JUN. (*Engineering*, 1928, 126, 219—220).—In the average rotary cement kiln, 200 ft. long and 9 ft. internal diameter, giving an output of 1100 tons of clinker per week from a slurry containing 38% of moisture and a coal of 12,600 B.Th.U./lb., the coal consumption is about 28% of the clinker produced and the temperature of the flue gases about 420—430° and of the clinker discharge about 310—320°. The heat introduced to such a furnace is distributed as follows: from the coal 92%, from the exothermic reaction 4.75%, and from the sensible heat of the constituents of the charge 3.25%. The heat expended is utilised as follows: in evaporating slurry water and superheating 40%, in decomposing carbonates 24.5%, in flue gases 20%, in clinker discharge 3%, and in radiation 9%, leaving 3.5% unaccounted for. A slight excess of air over the theoretical is necessary for economical working; too large an excess or a deficiency of air leads to a higher coal consumption. Accurate control of the kiln is obtained by installing recording pyrometers in the flues and clinker-discharge outlet, and

by frequent analysis of the flue gases, which should contain 1–2% of oxygen. A. R. POWELL.

Silicification of roads. G. LOPPENS and L. NYS (Bull. Soc. chim. Belg., 1928, 37, 263–266).—When sodium silicate solution is used as a bond for calcareous or granitic road materials, serious loss of binding power ensues if the mixture is kept before laying due to the action of atmospheric carbon dioxide in rendering up to two thirds of the silica insoluble. This may in part be prevented by using a silicate solution in which the ratio $\text{SiO}_2/\text{Na}_2\text{O}$ is less than 3. A. R. POWELL.

“Hiba” wood. UCHIDA.—See XX.

PATENTS.

Production of fused cement. SOC. ANON. ALUMINE ET DÉRIVÉS, and L. G. PATROUILLEAU (Addn. No. 31,931, 31.5.26, to F.P. 604,916; B., 1926, 880).—The fusion in the process described previously is effected inside a tubular arc furnace through which the material falls as it leaves the rotating furnace. The arc is struck between fixed and adjustable electrodes situated at the lower opening and within the furnace, respectively. L. A. COLES.

Preserving and protecting vegetable substances [wood]. L. P. CURTIN (B.P. 294,906, 31.1.27).—An impregnating solution for preserving wood contains a mixture of copper sulphate, sodium hydrogen sulphite, sodium carbonate, sodium chloride, arsenious oxide, and sodium fluoride in aqueous solution. The solution contains cuprous salts, which slowly oxidise in the wood with the deposition of insoluble cupric arsenite, which forms an effective fungicide. A. R. POWELL.

[Staining] treatment of wood. G. A. RICHTER, W. B. VAN ARSDEL, and J. G. GOSSELINK, Assrs. to BROWN Co. (U.S.P. 1,680,529, 14.8.28. Appl., 8.4.27).—Wood is heated above 100° to vaporise free moisture and expel air, and while hot is treated with gaseous ammonia under pressure. Partial condensation of the steam, *in situ*, takes place, and the ammonia enters and stains the wood, which is then removed. W. G. CAREY.

Manufacture of artificial stone. K. SCHENKEL (B.P. 285,470, 25.11.27. Ger., 19.2.27).—Porous artificial stone is made by forming a gel from an inorganic or organic material, *e.g.*, starch, by boiling or chemical treatment in a limited quantity of water, stirring the gel into a larger quantity of water, and adding hydraulic binding media, *e.g.*, calcined gypsum. W. G. CAREY.

Material for roofing, for paving streets, etc.
Roofing material. C. A. HOUQUES-FOURCADE (F.P. [A] 622,897—8, [B] 622,895, 9.10.26).—(A) Marine plants are boiled with aqueous solutions containing 5–10% of their weight of sodium carbonate, the solution, after removal of insoluble material, is treated with sulphur chloride or a solution containing it, and the pasty product is bleached by the addition of calcium chloride solution and, if necessary, milk of lime or a suspension of cement. The product is applied to felt, rolled, and dried, or two layers of it with an intermediate layer of rubber solution and sulphur are applied to the road surface and rolled with hot rollers. (B) Marine algae are ground with the gradual addition of dilute alkalis, treated with 5–10% of calcium oxide, cement, or

calcium chloride solution, and sieved. A mixture of the product (2 pts.) with cement (5 pts.) is applied to felt, rolled, and dried. L. A. COLES.

Preparation of emulsions or suspensions [for roads etc.]. G. BAUME, P. CHAMBIGE, and D. BOUTIER (B.P. 262,724, 9.8.26. Fr., 8.12.25).—The materials as described in B.P. 255,074 (B., 1928, 194) are prepared in concentrated form, the water or other liquid being added as and when required. W. G. CAREY.

Cold bituminous paving composition. O. H. BERGER (B.P. 293,830, 2.7.28. U.S., 13.7.27. Addn. to B.P. 257,114. Cf. U.S.P. 1,672,361; B., 1928, 672).—After mixing the asphalt cement and aggregate in the process described previously, about 3–5% of an “ameliorating agent,” *e.g.*, water and/or oil containing a considerable proportion of light oil, is added, and mixing is continued until the material assumes a light brown colour. L. A. COLES.

Proportioning the total water in concrete. C. S. JOHNSON (B.P. 295,894, 9.1.28).

Apparatus for making road compositions. L. ZACHARIAS and E. KLÜGLICH [OPPERMANN & DEICHMANN] (B.P. 289,066, 21.4.28. Ger., 22.4.27).

Cement from oil shale (U.S.P. 1,678,751).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Mechanism of the blast furnace, with especial reference to the combustion relations in the tuyère zone. W. LENNINGS (Arch. Eisenhüttenw., 1927—8, 1, 549–564; Stahl u. Eisen, 1928, 48, 1077–1079).—From the composition of the gases in the blast furnace in and just above the tuyère zone, it appears that with correct distribution of the tuyères there is an approximately ring-shaped zone, about 1 m. deep, in front of the tuyères in which all the oxygen of the blast is converted into carbon dioxide and ferrous oxide. The carbon dioxide rising through the hot coke is immediately converted into carbon monoxide, and the ferrous oxide unites with the slag, which travels downwards to the sump, where it is again reduced to metal by the coke which falls through the middle core inside the combustion ring. About 90% of the coke reaching the tuyère zone is gasified in the ring-shaped combustion zone, so that practically the whole of the blast-furnace charge passes down through the funnel-like aperture in the centre of this zone, in which the greater part of the heat of the furnace is produced; this explains the need of a sufficiently wide diameter at this part of the furnace in order to obtain a satisfactory output. Of the total oxygen content of the iron ore in the charge, 50% is removed by indirect reduction in the shaft, 15% by direct reduction in the lower part of the shaft, 13% by direct reduction in the boshes, and 20% by direct reduction in the sump. A. R. POWELL.

Theory of the blast furnace. G. EICHENBERG and P. OBERHOFFER (Arch. Eisenhüttenw., 1927—8, 1, 613–628; Stahl u. Eisen, 1928, 48, 1076–1077).—Analyses of the gases in five horizontal planes at varying heights in the iron blast furnace showed that the carbon dioxide content increased steadily from the walls towards the

middle, whilst the carbon monoxide content at first decreased, and then increased. This is ascribed to a loosening of the charge in an annular ring, midway between the walls and the centre, and to more vigorous reaction taking place in this section. As the charge in the outer zone sinks more slowly than that in the middle, the so-called "reduction limit" of the iron oxide occurs earlier here, and a heavy deposit of carbon is formed by the decomposition of carbon monoxide; this accounts for the characteristic rise in temperature observed in this zone. Experiments with charges in which all the carbon dioxide was added as limestone, and with others in which part of the limestone was replaced by minette, showed that practically all the limestone passed unchanged to the lower parts of the furnace, whereas the iron carbonate was rapidly decomposed in the upper parts.

A. R. POWELL.

Siemens basic steel utilising pig iron from South African ores. G. M. NODEN (J. S. African Chem. Inst., 1928, 11, 41—54).—A brief description of the Newcastle steelworks (S. Afr.) with analyses of raw materials and finished products obtained from basic, open-hearth, steel furnaces operating on pig iron obtained from South African spathic iron ore and on scrap obtained from the railways and mines. Tabulated details of the construction and working of four furnaces and of the necessary gas producers are also included.

A. R. POWELL.

Eighth report to the Corrosion Research Committee of the Institute of Metals. Corrosion of condenser tubes. "Impingement attack"; its causes and methods of prevention. R. MAY [with H. C. H. CARPENTER] (Inst. Metals, Sept., 1928. Advance copy. 35 pp.; cf. B., 1924, 833).—In an introduction Carpenter summarises the previous work of the Committee.

I. The formation and breakdown of the protective films formed on condenser tubes or other metal specimens can be studied conveniently by measuring the potential changes corresponding to the changes in the film. A suitable apparatus using a three-electrode valve potentiometer is described. The "film potential" is defined as the electrode potential of the clean metal minus the potential of the film-covered metal. A clean 70:30 brass tube (containing 0.02% As) was tested with a special jet apparatus using sea water, and the gradual formation of the protective film was detected by a rise in the film potential, which reached a steady value. On scratching the film, the film potential at once sank, and the scratch was not completely healed after 24 hrs., whilst if air was admitted the incompletely healed film at once broke down and violent impingement attack occurred. But with a special brass tube (Cu 74.64%, Al 1.98%, Fe 0.13%, Zn remainder) under similar conditions the scratch was completely healed in 8 hrs., and the admission of air had no effect.

II. Tests with an experimental condenser showed that impingement attack could take place in the presence of air bubbles alone, and without the intermittent cavitation which Parsons has shown (Proc. Inst. Naval Architects, 1927) to be responsible for the phenomenon in some cases. Very small bubbles are comparatively

harmless, but experiments with a jet-test apparatus—in which a jet of sea water containing air impinged on the specimens—showed that with 70:30 brass impingement attack always occurred if the air bubbles were violently broken up into smaller bubbles where the water impinged on the metal, the velocity of the water which contained 1% (by vol.) of air being greater than 6 ft./sec.

III. Rotating motion of the water in a condenser is particularly harmful because it causes the air bubbles to coalesce. A wire grid may serve to break up the large bubbles into comparatively harmless smaller bubbles, but correct design is considered more important. Two alloys specially resistant to impingement attack are 70:30 cupro-nickel and the aluminium-brass described above.

W. HUME-ROTHERY.

Corrosion at discontinuities in metallic protective coatings. U. R. EVANS (Inst. Metals, Sept., 1928. Advance copy. 33 pp.).—I. Samples of very mild steel were coated by spraying with copper, zinc, nickel, or aluminium. The coatings were either (1) intentionally discontinuous, (2) thin (0.001 cm.), continuous, but porous, (3) thicker (0.01 cm.) on clean metal, or (4) similar to (3) but on greasy metal. The corrosion of these samples, in both bent and unbent conditions, was then studied in (a) partial immersion in 0.5*N*-sodium chloride, or Cambridge water (a typical hard water), (b) intermittent spraying with 0.5*N*-sodium chloride or 0.01*N*-sulphuric acid, (c) exposure to air containing sulphur dioxide and moisture, or hydrochloric acid and moisture. Cracks produced by bending are more dangerous than pores distributed more or less uniformly. If the coating metal is cathodic to steel (*e.g.*, copper or nickel) the steel is corroded at the crack, and marked acceleration of corrosion is caused by copper, nickel being less dangerous. With zinc the coating suffers corrosion and protects the steel; thick coats are here better than thin in spite of the greater tendency to crack. Aluminium, which is less strongly anodic than zinc, gives better protection than zinc in the sodium chloride solution since it protects the iron without being violently attacked; but in Cambridge water aluminium is worse than zinc because the rate of attack of aluminium is too slow for it to exert its protective effect. In the spray tests both copper and nickel give good protection. In immersed corrosion rust is precipitated outside the coating, but in atmospheric or spray-test corrosion the rust is formed below the coating.

II. Similar tests were made with steel coated with zinc by (a) spraying, (b) electrodeposition, (c) thick and thin sherardising, and (d) hot dipping. Both (c) and (d) give zinc-iron alloys in the coating, and these are less readily attacked than free zinc, but for this reason give less sacrificial protection to the steel in some waters. Alternate spraying and drying with the chloride or sulphuric acid solutions builds up a film on all the zinc coatings which greatly increases the resistance to corrosion by chloride solutions.

W. HUME-ROTHERY.

Phenomena of corrosion of iron and steel. A. HERRERO and M. DE ZUBIRIA (Iron and Steel Inst., Sept., 1928. Advance copy. 16 pp.).—A discussion and review of modern theories of the mechanism of

corrosion of iron and steel, with some suggestions for reducing the large annual loss of these metals by corrosion.

A. R. POWELL.

Tempering of hardened steels and influence of silicon and nickel on the process. H. BIRNBAUM (Arch. Eisenhüttenw., 1928-9, 2, 41—47; Stahl u. Eisen, 1928, 48, 1125—1126).—Tempering at 180° of hardened steels containing more than 0.33% C results in a contraction in volume which increases linearly with the carbon content; this is ascribed to the decomposition of a martensitic phase containing more than 0.33% C (cf. Hanemann and Traeger, B., 1926, 982). At slightly higher temperatures a second contraction takes place, the magnitude of which is also a linear function of the carbon content; this contraction begins with steels containing more than 0.115% C, but the curve shows a change of direction at 0.33% C such that the sum of the two contractions in steels containing more than 0.33% C lies on the extension of the straight line showing the contraction/carbon content for steels containing 0.115—0.33% C. Hardened steels with less than 0.115% C do not contract on tempering as the cementite is liberated from solid solution without change of volume. It is concluded that ϵ -martensite contains a maximum of 0.115% C and η -martensite more than 0.33% C, the phase with 0.11—0.33% C being a previously unrecognised fourth variety of martensite. Nickel has no effect on the volume changes on tempering except in so far as it causes the retention of austenite in quenched high-carbon steels; silicon raises the temperature at which the second contraction takes place.

A. R. POWELL.

Influence of pearlitisation below the A₁ point on the mechanical properties of carbon steels. J. ORLAND (Iron and Steel Inst., Sept., 1928. Advance copy. 8 pp.).—Tempering of quenched hypoeutectoidal steels at a temperature slightly above that at which Hallimond's "labile shower" (B., 1922, 418 A) takes place produces a finely-granulated pearlite which imparts to the steel a higher tensile strength and impact resistance than that produced by laminated pearlite in ordinary annealed steel. For mild steels the best tempering temperature appears to be 500°, and for steels containing more than about 0.5% C tempering at 580° gives the best results.

A. R. POWELL.

Influence of varying strains and annealing temperatures on growth of ferrite crystals in mild steel. C. A. EDWARDS and T. YOKOYAMA (Iron and Steel Inst., Sept., 1928. Advance copy. 23 pp.).—Tests on sheets and bars of mild steel (0.08% C) strained in a Buckton tensile machine and subsequently annealed at 500—725° showed that there is a critical strain for maximum crystal growth for every annealing temperature, the strain-temperature graph being a straight line. The higher the annealing temperature, provided the straining corresponds with the critical degree, the larger are the crystals and the longer is the time required for maximum growth, but with high annealing temperatures the material is more sensitive to small variations of strain. Strains below the critical amount do not cause crystal growth on annealing, and strains above the critical amount produce crystals

which are smaller the greater is the strain above the critical, the strain-growth graph being a hyperbola. Maximum grain size of this steel is obtained by annealing at the A₁ point (about 725°) after straining to produce an elongation of 4%. Very large crystals cannot be grown owing to the retarding effect of the pearlite present which migrates to the crystal boundaries after annealing at the A₁ point. Annealing at 675—700° causes the separation of spheroidised cementite, and annealing at 870° the formation of massive cementite frequently in the form of a horse-shoe pattern. If columnar crystals develop in the outer parts of the specimen during annealing the central parts show no signs of crystal growth; conversely, when crystal growth takes place in the centre no columnar crystals form in the outer parts. The growth of these columnar crystals is due to the combined influence of cold-work and decarburisation; e.g., at 725° large columnar crystals grow in specimens strained 4—6%, but not in unstrained specimens, if annealing is conducted under decarburising conditions. The higher the annealing temperature under these conditions the greater is the amount of strain which is neutralised by the development of columnar crystals at the rim and the greater is the strain required to produce large crystals in the centre. When decarburisation occurs there is a tendency for the cementite to migrate to the crystal boundaries of the new ferrite grains and there form a thin film which decreases the strength of the steel.

A. R. POWELL.

Tensile test of steels at high temperatures. T. INOKURY (Sci. Rep. Tōhoku Imp. Univ., 1928, 17, 791—816).—The tensile strength, elongation, and reduction in area were determined for Armco iron and steels containing 0.1—1.5% C at 20—1100°. The tensile strength decreases up to 100° and then increases to a maximum value at 300°. It falls gradually with increasing temperature up to 850°, rises to a maximum at 930°, and then falls steadily at higher temperatures. These changes are most marked with low-carbon steels, and the temperatures at which they occur fall with increasing carbon content. The elongation and reduction in area show similar changes, but in the opposite sense, at the same temperatures. It is concluded that carbon steels should be forged above 1000°, when the plasticity is a maximum.

C. J. SMITHELLS.

Change in tensile strength due to ageing of cold-drawn iron and steel. L. B. PFEIL (Iron and Steel Inst., Sept., 1928. Advance copy. 15 pp.).—The alteration of the tensile strength of cold-drawn steel, employed as a measure of the capacity of the metal to age-harden, showed that the presence of carbon was the principal factor. The average increase in tensile strength due to ageing cold-drawn mild steel (0.11—0.14% C) for one month amounted to 2.32 tons/in.², and only when the carbon content was below 0.0025% was the metal incapable of hardening. The structural state of the carbon was of negligible importance, and it is considered that the ageing effect is due solely to the carbon in solution in ferrite, and that the solubility of carbon in cold-worked ferrite is less than in annealed ferrite. Ferrite which had been exposed to reducing conditions (hydrogen) and which had no ageing

properties was found to regain these when subjected to oxidising conditions, and it is shown that the presence of oxides in the metal produced by diffusion of oxygen caused age-hardening. C. A. KING.

Properties of cold-drawn and of heat-treated steel wire. S. H. REES (Iron and Steel Inst., Sept., 1928. Advance copy. 16 pp.).—The effect of heating the cold-drawn material is, in general, to increase the density, an increase of 0.33% being observed when vanadium-chromium steel was heated to 750°. Elasticity at atmospheric temperature was restored completely to this type of steel when heated to 300° or above as compared with 200° for mild steel wire, and other changes which occur on low-temperature heat-treatment of mild steel are comparable in extent when the chromium-vanadium steel has been heated at about 100° higher. The superiority of the latter steel treated for 1 hr. at 400° over the most suitably treated cold-drawn carbon steel wire (1 hr. at 300°) is considerable. C. A. KING.

Effects observed in quenched liquid steel pellets and their bearing on bath conditions. J. H. WHITELEY (Iron and Steel Inst., Sept., 1928. Advance copy. 13 pp.).—As an aid in judging the progress of refining of a bath of steel, the microscopical examination of pellets of the steel chilled in cold water is suggested. Distribution of sulphide inclusions was found to be by no means uniform, the inference being drawn that the solubility of sulphide in liquid steel is extremely low. Oxide particles, usually associated with the sulphide, occur if the steel is not completely deoxidised, and tend to increase as the carbon content diminishes below 0.2%. The presence of minute spherical cavities due to gas bubbles generally indicates good conditions in the metal, the number of such inclusions increasing with the refinement of the steel. The cellular network observed on etching is due probably to the heterogeneous nature of the molten metal, and although the definite causes of this have not been ascertained, the presence of oxygen does not appear to be essential as the most defined effects are to be observed in thoroughly deoxidised steels. C. A. KING.

Use and interpretation of the transverse test for cast iron. J. G. PEARCE (Iron and Steel Inst., Sept., 1928. Advance copy. 18 pp.).—Attempts to correlate the strength with size and composition of cast iron showed that the transverse rupture modulus increases as the test-bar diameter diminishes, until the metal ceases to be grey. The size-strength curve is a useful index to the behaviour of cast iron in thick sections, and the relationship between the transverse strength and the tensile and compressive strengths is of value only if expressed as rupture modulus. C. A. KING.

Mechanism of carbon penetration in cementation of iron and steel. G. TAKAHASHI (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 761—782).—The cementation of iron and steel by carbon monoxide was studied by following the changes in pressure inside a steel tube during carburisation by carbon monoxide, and decarburisation by carbon dioxide, circulating outside the tube. The weight of carbon taken up by the iron is much greater than could result from the diffusion and

subsequent decomposition of carbon monoxide, taking the accepted values for its solubility. The assumption that carbon deposited on the surface of the metal diffuses by means of carbon oxides present in the metal is untenable for the same reason. It is concluded that carburisation results from the diffusion of nascent carbon formed by the decomposition of the gas at the surface of the metal. C. J. SMITHELLS.

X-Ray investigation of the formation of martensite. K. HONDA and S. SEKITO (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 743—760).—X-Ray spectrograms of a series of quenched carbon steels show that the outer layers of the specimens contain a body-centred tetragonal lattice, and the inner portion a body-centred cubic lattice. These are identified with α - and β -martensite, respectively. Austenite was always found intermixed with martensites, the concentration decreasing towards the centre of the specimen. The formation of martensites from austenite is explained on the assumption of a uniform compression in the tetragonal axis of the austenite, and a uniform expansion in the perpendicular direction. The broadening of the spectral lines in martensite is attributed to strain in the iron lattice caused by the presence of carbon atoms in its interspace. C. J. SMITHELLS.

Accurate gas burette for the volumetric determination of carbon [in steel]. O. MEYER (Chem. Fabr., 1928, 520—521).—A number of determinations can be carried out under the same pressure and temperature by the use of an auxiliary manometer terminating in a bulb within the cooling jacket of the burette. The air enclosed in this bulb is brought to the desired pressure once for all by the use of a second manometer. Efficient water circulation in the jacket provides for temperature control. C. IRWIN.

Resistance of pure chromium-nickel-iron alloys to corrosion by acids. W. GUERTLER and W. ACKERMANN (Z. Metallk., 1928, 20, 269—279).—The rate of dissolution of pure homogeneous solid-solution alloys of iron, nickel, and chromium has been determined in 1 and 20% nitric acid and in 1% sulphuric acid; in general, the results confirm Tammann's $m/8$ law. Thus the rate of dissolution of nickel-iron alloys in nitric acid increases suddenly with 12.5 at.-% Ni, and decreases again suddenly at 25 at.-% Ni; and chromium-iron alloys become passive when the chromium content exceeds 12.5 at.-%. In the ternary alloys passivity towards nitric acid is afforded by a similar proportion of chromium, variations in the nickel and iron content having no effect on the behaviour of the alloys. Iron-nickel alloys have a higher resistance to corrosion by sulphuric acid when the nickel exceeds 12.5 at.-%, whereas chromium-iron alloys dissolve more rapidly when the chromium exceeds 12.5 at.-% and most rapidly when it exceeds 25 at.-%. In the ternary alloys the molecular limit 2/8 (Cr+Ni) seems to be of importance especially when the chromium content is high, e.g., 17—18% Cr and 6% Ni; this alloy is fairly resistant to attack by 1% sulphuric acid, whereas alloys containing more than 75 at.-% Fe are more readily dissolved than is iron itself. Towards acetic acid and mixtures of hydrochloric acid and hydrogen peroxide excess of chromium above 12.5

at.-% induces passivity. The above results indicate that chromium alone has a protective action when an oxidising agent is present and nickel alone in the absence of an oxidising agent.

A. R. POWELL.

Thermal and microscopical investigation of alloys of reactive metals. W. HUME-ROTHERY (Inst. Metals, Sept., 1928. Advance copy. 16 pp.).—I. Methods suitable for these investigations are described and criticised under the following headings: (1) cooling-curve methods; (2) refractories; (3) difficulties in determining the composition of the cooling curve ingot at the moment of solidification; (4) preparation of chill castings; (5) microscopic methods. II. The method of determining the composition of a phase from the duration of the arrests on the cooling curves is sound in the case of simple eutectic arrests where no solid solutions are present. But in the case of peritectic reactions or where solid solutions are formed, the method is unsound at all except the highest temperatures. III. The best methods for use where super-cooling or suspended transformation is met with are discussed.

W. HUME-ROTHERY.

Thermal brittleness in metals. T. INOKUTY (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 817–842).—Assuming that the increase in tensile strength of steels at 200–300° results from work-hardening, a similar effect was looked for in other metals. It is concluded from measurements of tensile strength and reduction in area between –190° and +200° that thermal brittleness is shown by aluminium, tin, and lead between –100° and 40°, by zinc at 120°, and by copper and brass at 100–500°.

C. J. SMITHELLS.

Shrinkage and surface stresses in hard-drawn copper, aluminium, bronze, aldrey, and aludur wires. H. BOHNER (Z. Metallk., 1928, 20, 286–288).—On heating hard-drawn wires of the above-named metals the primary expansion due to increase of temperature is replaced at a certain temperature, depending on the metal and on the rate of heating, by a contraction the magnitude of which is to a large extent dependent on the time of heating and the maximum temperature reached. The temperature range at which contraction commences is 200–230° for aluminium, about 250° for aludur and aldrey, 400–600° for copper, and 350–500° for bronze. The electrical conductivity of the wires follows the expansion, an increase taking place up to the temperature at which contraction begins followed by a gradual decrease during the period of contraction owing to the development of a coarsely crystalline structure and the formation of voids between the crystals caused by the contracting force of the surface stress overcoming the cohesive force between the crystals.

A. R. POWELL.

α -Phase boundary of the copper-silicon system. C. S. SMITH (Inst. Metals, Sept., 1928. Advance copy. 12 pp.).—The α -phase boundary of this system has been determined by annealing and quenching experiments supplemented by thermal analysis. In the region in which the α -solid solution is in equilibrium with liquid, the solid solubility limits increase from 0 at the m.p. of copper to 5.25% Si at 852°, at which temperature the α -solid solution reacts with liquid to form a new phase (β). As the temperature falls from 852° to 782° the

α -solid solubility limit increases from 5.25% to 6.7% Si, remains approximately constant from 782° to 721°, and then gradually decreases until at 400° it is only 4.1% Si. These variations correspond to transformations of the β -phase (in equilibrium with the α) at 782° and 721° of which the nature is undetermined, although the former is probably eutectoidal.

W. HUME-ROTHERY.

Copper-magnesium alloys. III. W. R. D. JONES (Inst. Metals, Sept., 1928. Advance copy. 11 pp.; cf. B., 1926, 830; 1927, 817).—Notched-bar impact tests have been carried out on forged and heat-treated copper-magnesium alloys containing up to 10% Cu. The tests were carried out in a 30-kg. Charpy machine using the B.E.S.A. standard form of test piece. Simple heat-treatment has no beneficial effect on forgings. The addition of 1.5% Cu to magnesium raises the impact value from 8.3 to 14.3 ft.-lb., but no advantage is gained in adding more than 2% Cu, whilst with 5% Cu the alloys become brittle, the brittleness being less if the temperature of the test is raised; the tests were carried out at temperatures up to 275°. On keeping for 20 days at –7° and testing at this temperature, the impact values are decreased, and brittleness is caused.

W. HUME-ROTHERY.

Constitution of the alloys of aluminium with copper, silicon, and iron. A. G. C. GWYER, H. W. L. PHILLIPS, and L. MANN (Inst. Metals, Sept., 1928. Advance copy. 52 pp.).—I. Recent work on the binary alloys of aluminium with copper, silicon, and iron is reviewed. II. (a) The ternary system aluminium-copper-silicon has been investigated in the range 0–40% Cu and 0–20% Si. Within this range the alloys form a simple eutectiferous ternary system, the constituents being the compound CuAl_2 , aluminium containing some copper and silicon in solid solution, and silicon. The ternary eutectic point is at 525° and contains 26.0% Cu, 6.5% Si, and 67.5% Al. (b) The ternary system, aluminium-copper-iron has been investigated over the range 0–40% Cu, and 0–10% Fe. The constituents are an aluminium-rich solid solution, the compounds FeAl_3 and CuAl_2 , and an iron-rich ternary constituent "N" (see III below). This constituent is formed at 590° by a peritectic reaction between FeAl_3 and liquid. Aluminium, CuAl_2 , and "N" form a ternary eutectic at 32.5% Cu, 0.3% Fe, and freezing at 540°. (c) Recent work on the ternary alloys of aluminium, silicon, and iron is reviewed. III. The quaternary system, aluminium-copper-silicon-iron has been investigated over the range 0–40% Cu, 0–15% Si, and 0–10% Fe. The constituents present are those of the respective ternary systems, and no additional or true quaternary constituents are formed. The alloys contain a quaternary eutectic consisting of aluminium, silicon, CuAl_2 , and the "X" constituent of the aluminium-silicon-iron system, the eutectic composition being 26% Cu, 6.5% Si, and 0.5% Fe, and freezing at 520°. The structure of the quaternary alloys indicates that the "X" constituent of the aluminium-silicon-iron system, and the "N" constituent of the aluminium-copper-iron system are the end members of a continuous series of solid solutions, varying from a hard, white, dark-etching, silicon-rich form to a soft, pinkish, light etching, copper-rich form. The paper deals exclusively with the structure of

the alloys as ordinarily prepared by rapid or slow cooling.

W. HUME-ROTHERY.

Die-casting of copper-rich alloys. R. GENDERS, R. C. READER, and V. T. S. FOSTER (Inst. Metals, Sept., 1928. Advance copy. 32 pp.).—Die-casting tests on various aluminium bronzes with and without the addition of iron, manganese, nickel, or lead have been made in commercial moulds with satisfactory results as regards tensile strength, hardness, and surface qualities. The mechanical properties of the best alloys for this purpose are tabulated. Aluminium bronzes (59.9% Cu, 3.5% Al, remainder zinc, and 69.2% Cu, 4.3% Al, remainder zinc), have excellent properties for die-casting purposes; and they have a higher proof stress, a smaller attack on the moulds, and are cheaper than aluminium bronzes. Tests on a large number of ferrous alloys for use in the manufacture of die-casting moulds showed that high-carbon steels, special heat-resisting steels, and cast iron low in phosphorus are satisfactory for this purpose, whereas low-carbon steels and engineering steels are rapidly attacked by the molten alloy. Chromium plating and calorising afford efficient protection to almost all ferrous alloys used in the manufacture of moulds.

A. R. POWELL.

Silicon-aluminium casting alloys. J. DORNAUF (Z. Metallk., 1928, 20, 289–292).—The maximum strength of modified silicon-aluminium alloys is obtained with the eutectic composition, and with correct heat-treatment a tensile strength of 18–23 kg./mm.² may be obtained. This is increased slightly by the addition of 0.8% Fe, but is rapidly decreased by a larger proportion of iron and by addition of even very small amounts of magnesium. Manganese up to 1% has little effect on the mechanical properties; larger quantities increase the hardness, but reduce the tensile strength and ductility. More than 1% of copper or zinc or more than 0.05% of tin or antimony causes a serious decrease in tensile strength and ductility; copper and antimony increase the hardness, whereas tin and zinc decrease it. On account of its relatively high resistance to corrosion compared with that of other aluminium alloys, silumin forms a suitable material for making castings for the chemical industry, e.g., evaporators.

A. R. POWELL.

Properties and production of aluminium alloy die-castings. S. L. ARCHBUTT, J. D. GROGAN, and J. W. JENKIN (Inst. Metals, Sept., 1928. Advance copy. 19 pp.).—The properties and production of die-castings in permanent metal moulds under gravity feed have been determined using the following aluminium alloys: 4% Cu, 8% Cu, 12% Si, 4% Cu + 3% Si, and Y-alloy. The test pieces were of two shapes—a cylindrical rod with shoulders for direct tensile tests, and a hollow tubular casting requiring the use of cores in moulding and incorporating flanges and sections of varying thickness. The moulds were dressed with a suspension of whiting in sodium silicate, and the cores with a paste of plumbago and rouge. The rod castings were most satisfactory when made in a horizontal mould with a runner and riser placed symmetrically over the two heads of the test pieces. To obtain satisfactory tubular castings a set of conditions had to be worked out for every alloy, the following points having to be standardised:—casting

and mould temperatures, rate of pouring, and interval between filling the mould and drawing the cores, opening the mould and stripping. The hot-shortness of the above five alloys has been determined by measuring the energy absorbed in the fracture of an unnotched test-bar cast to shape. The impact strength of all the alloys remains high until about 5° below the m.p. in the case of the 12% Si alloy to about 45° below the m.p. in the case of the 3% Si + 4% Cu alloy; above these temperatures it rapidly falls to zero.

A. R. POWELL.

[Zinc] die-casting alloys of low m.p. T. F. RUSSELL, W. E. GOODRICH, W. CROSS, and N. P. ALLEN (Inst. Metals, Sept. 1928. Advance copy. 15 pp.).—The tensile strength, bending strength, hardness, density, and porosity of sixteen zinc-base, pressure-cast alloys hardened with copper-tin or copper-aluminium have been determined with especial reference to the influence of casting temperature, mould temperature, size of gate and vent, and type of pull on the lever of the testing machine. The last-named variable has the most pronounced effect on the porosity, but the differences tend to decrease as the size of gate opening is increased. The casting and mould temperatures have only a small effect on the tensile strength compared with the effect of the form of the test-piece and of non-axial loading; alloys with a copper-aluminium hardener are almost twice as strong as those with one of copper-tin. The initial contractions of different castings of the same shape and of the same alloy may vary by as much as 50% according to the pull exercised on the plunger in casting, and, in general, alloys with aluminium contract more than those containing tin. The rate of growth on ageing is the same for castings of both series of alloys of the same form and porosity; in hot air the rate of growth is more rapid than in cold air, but the magnitude of the growth is the same. In steam, however, growth is much greater due to chemical action. Some tests on the action of molten zinc on steels of varying composition showed that calorising prevents entirely the attack of zinc, but that nickel- or chromium-plating are useless.

A. R. POWELL.

Work-softening of eutectic alloys. F. HARGREAVES and R. J. HILLS (Inst. Metals, Sept., 1928. Advance copy. 14 pp.).—Micrographic examination and Brinell hardness tests of the lead-tin eutectic alloy after reduction in thickness by hammering indicate that there is a critical point at 30% reduction. Although both tin and lead recrystallise spontaneously after about 40% reduction in thickness by hammering, the eutectic shows a marked retardation in recovery from work due to the presence of two phases, and this lag is the greater the finer the subdivision of the phases. Cold-hammering of the recrystallised eutectic alloy causes a marked reduction in hardness, which is ascribed to interphase movement and lag in recovery from work, but is not a peculiarity of the eutectic structure, as tin containing 15% Pb behaves similarly; tin with 0.5% Pb may also be rendered softer by working the cast alloy. The hardness of cast and of worked and annealed zinc-cadmium eutectic is decreased by cold-work, but the original hardness is recovered quickly and almost completely on keeping at the ordinary temperature.

A. R. POWELL.

Relation between pressure and diameter of impression in hardness test. K. TAKAHASHI (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 843–856).—The relation between the pressure P and the diameter of indentation d in the Brinell test is given by $P = ad^n$ for large loads, a and n being constants characteristic of the substance. For pressures of 5–100 kg., a and n decrease with the pressure, and the value of n approaches 2 as the pressure decreases. C. J. SMITHELLS.

Induction furnace. KRAEMER.—See XI.

PATENTS.

Cupola furnaces. E. PIVOVARSKY (B.P. 271,910, 31.5.27. Ger., 31.5.26).—To diminish the absorption of carbon in the production of iron alloys poor in carbon (about 1%), the initial charge of coke on the hearth of the furnace is replaced wholly or partially by pieces of graphite. The crucible or hearth of the furnace is in the shape of an inverted cone so that the duration of contact of the graphite charge with the descending molten charge is reduced. M. E. NOTTAGE.

Cupola. A. E. BAILLOT, Assr. to A. H. and A. H. COPLAN (U.S.P. 1,678,899, 31.7.28. Appl., 5.3.26).—The refractory lining of the middle part of a cupola is replaced by a metal cylinder provided with a series of parallel annular flues through which the air-blast to the cupola is passed in order to preheat it. The discharge from these flues is passed directly to the furnace through a conduit which connects the flues with an air-belt feeding the tuyères. A. R. POWELL.

Preparing flue dust for smelting in blast furnaces. F. F. MARQUARD (U.S.P. 1,680,107, 7.8.28. Appl., 15.10.27).—Iron ore flue dust is mixed with ammonia-still waste liquor. H. ROYAL-DAWSON.

Annealing [of metals]. C. R. G. STEWART, Assr. to WESTERN ELECTRIC CO. (U.S.P. 1,673,271, 12.6.28. Appl., 8.5.25).—The metal articles to be annealed are packed in a metal container between layers of powdered electrolytic iron containing occluded hydrogen, and the container is closed and heated to the desired temperature. During this operation the hydrogen liberated from the iron prevents oxidation of the metal being annealed. A. R. POWELL.

Treatment of metallic [iron] ores, or other metallic products. T. ROWLANDS (B.P. 295,338, 10.2.27).—Iron ore is preheated in the first chamber of a three-chamber rotating cylindrical furnace, the hot ore is mixed with pulverised coke from a low-temperature carbonisation plant and passed into the second chamber, heated externally and internally by the gases from the carbonisation plant and supplied with sufficient carbon monoxide to effect complete reduction of the iron oxide to metal, and the hot gases are utilised in heating the retorts of the carbonisation plant and for re-conversion into carbon monoxide for use again in the process. The reduced iron passes into the third chamber, where it is cooled and the heat utilised for coking further quantities of coal. The reduction is effected at 500–900°, preferably at 700–800°, and the gases from the coking plant are stripped of their valuable by-products prior to use in the reduction furnace. A. R. POWELL.

Production of wrought iron direct from electrolytic iron. R. D. PIKE (U.S.P. 1,675,867, 3.7.28. Appl., 30.6.24).—Wrought iron composed of laminae of electrolytic iron interspersed with laminae of slag is claimed. A. R. POWELL.

Production of iron castings. K. SIPP, Assr. to H. LANZ A.-G. (U.S.P. 1,678,655, 31.7.28. Appl., 3.11.25. Ger., 30.11.24).—Grey iron castings of uniform structure and hardness throughout are obtained by casting a suitable iron into moulds under such conditions that the rate of cooling in certain thick sections is adapted to produce a workable grey iron, whereas the rate of cooling in thicker sections is accelerated and in thinner sections retarded. A. R. POWELL.

Pickling of iron etc. O. VOGEL (B.P. 277,308, 12.8.27. Ger., 8.9.26).—One or more colloids, e.g., sulphite-cellulose waste-liquor, glue, agar-agar, molasses, etc., are added to pickling baths in addition to hydrocarbons, compounds containing a quinoline nucleus, etc. as described previously (cf. B.P. 158,768, 188,718; B., 1921, 223 A; 1923, 20 A). L. A. COLES.

Magnetic [iron] alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 295,443, 13.5.27).—Magnetic alloys which can readily be powdered at the ordinary temperature consist of 85–95% Fe with 1–0% Cr and 1–10% Si, the latter being 3–5 times the chromium content. The alloys are substantially free from carbon and manganese, and are deoxidised by 0.1–0.3% Al. A. R. POWELL.

Heat-resisting alloy. A. L. BOEGEHOLD, Assr. to GEN. MOTORS RES. CORP. (U.S.P. 1,680,007, 7.8.28. Appl., 6.6.27).—An iron alloy contains 2.4–2.8% C, 1.75–2.25% Si, 3.75–4.25% Ni, and 5.75–6.25% Al. F. G. CROSSE.

Steel alloy. G. L. NORRIS, Assr. to VANADIUM CORP. OF AMERICA (U.S.P. 1,680,301, 11.8.28. Appl., 19.1.27).—The alloy contains 0.4–2% Si, 0.05–0.5% V, 0.2–0.6% Mn, and 0.1–1% C, the balance being iron with the usual impurities of commercial steel. F. G. CROSSE.

Alloy steel. H. B. ALLEN, Assr. to H. DISSTON & SONS, INC. (U.S.P. 1,681,797, 21.8.28. Appl., 27.4.25).—A high-carbon tool steel which is alloyed with 1–1.5% Ni and 0.1–0.7 Mo. F. G. CROSSE.

Steel alloys possessing high strength at high temperatures. F. KRUPP A.-G. (B.P. 284,314, 6.1.28. Ger., 28.1.27).—The alloys contain 8–18% Cr, 2–12% Mn, 3–12% W, up to 1.2% Si, and up to 1% C. A particular alloy contains 12–14% Cr, about 5% Mn, about 5% W, and about 0.5% C, together with the usual admixtures of silicon, sulphur, and phosphorus. F. G. CROSSE.

[Roasting furnace for] treatment of ores etc. and metallurgical products. F. L. WILDER, E. MORRIS, E. SCHIFF, and E. S. KING (B.P. 295,226, 6.1.27).—A furnace for roasting ores etc. comprises a vertical tubular muffle surrounded by an annular combustion chamber fed with producer gas and preheated air, and surmounted by a continuous feeding device. The hot roasted product discharges through a reciprocating door into a conical pit through which the air required for oxidising the ore and for burning the producer gas

passes and is preheated. Further preheating of the combustion air is effected by allowing it to pass upwards through an annular channel surrounding the combustion chamber before it enters that chamber at the upper end.

A. R. POWELL.

Method of roasting and reducing ores. B. CHRISTIANSEN (U.S.P. 1,680,861, 14.8.28. Appl., 29.3.26. Swed., 7.4.25).—The ore is passed through the preheating, roasting, prereluction, and final reduction zones of a channel furnace. In the last zone it is treated with circulating reducing gases, which are recarbonised in alternatively operating recarbonising furnaces, the temporarily inoperative one of which is reheated by partial combustion in it of the excess of circulating gases. The products of the partial combustion are supplied to the prereluction zone, and finally burned in excess air in the roasting zone.

B. FULLMAN.

Treatment of ores, residues, etc. for extracting metal [copper, lead, and zinc] values. S. C. SMITH (B.P. 295,224, 5.4.27).—The ore is leached with dilute nitric acid, and the filtered solution treated with milk of lime to precipitate the metals dissolved; in case several metals are present, *e.g.*, iron, copper, zinc, the precipitation may be carried out fractionally, using limestone to precipitate the iron, and a deficiency of milk of lime or zinc oxide to precipitate the copper. The calcium nitrate solution is either evaporated to obtain a fertiliser salt or treated with sulphuric acid for the regeneration of nitric acid. When the presence of calcium sulphate in the residue from the leaching operation is unobjectionable, *e.g.*, in leaching lead-zinc fume, the leaching may be carried out with a mixture of sulphuric acid and calcium nitrate solution from a previous operation.

A. R. POWELL.

Treatment of ores etc. W. VAN REES (F.P. 623,445—6, 17.2.26).—Ores are reduced, chlorinated, etc. in a preheating furnace of the rotating tube type in which they may be heated until fusion begins, and the process is completed in an adjoining swinging furnace. The waste gases and gaseous products from the first furnace are used for heating the second, and those from the second are used for heating the first. The furnaces are connected with two recuperators used alternately.

L. A. COLES.

Metallurgical process. SOC. ANON. LE NICKEL (F.P. 623,494, 23.2.26).—Fused metals are atomised by a current of gas. The process can be used, *e.g.*, for decarbonising and for the removal of iron from ferro-nickel, ferrochrome, etc., oxidising gases being used and the product being fused with material capable of forming a slag with the iron oxide formed.

L. A. COLES.

Metallurgical process [for obtaining metals from oxides, sulphides, etc.]. VACUUMSCHMELZE GES.M.B.H., H. GRUBER, W. ROHN, and O. H. WEBER (B.P. 294,795, 22.9.27).—Oxides or sulphides of metals are heated in an induction furnace at a pressure below the dissociation tension of the compounds at the temperature employed whereby oxygen or sulphur are evolved and a residue of pure metal is left; the latter may often be melted by increasing the temperature. Thus copper oxide yields copper and oxygen at 1090° under 7 mm., nickel oxide

dissociates to metal and oxygen at 1450° under 6 mm., molybdenite yields molybdenum sponge and sulphur at 1400° under 2.4 mm., and tungsten trioxide yields tungsten powder at 1000° under 2 mm. pressure. The process is adapted to the preparation of alloys and to the removal of traces of oxygen and sulphur from metals, *e.g.*, copper or nickel, as well as to the removal of oxide skins from the surfaces of worked and heat-treated metals.

A. R. POWELL.

Producing metals from their sulphides in electric furnaces. E. G. T. GUSTAFSSON (B.P. 280,540, 7.11.27. Swed., 11.11.26).—A mixture of a sulphide ore of a volatile metal and metallic iron in the form of sponge or small pieces the size of a pea or nut is fed on to the slag layer of an electric furnace of the combined arc and resistance type whereby the volatile metal is expelled and an iron or iron-copper matte carrying the precious metals is formed.

A. R. POWELL.

Manufacture of sound ingot and other metal castings. K. HONDA (B.P. 281,992, 10.6.27. Japan, 9.12.26).—The main casting mould is filled up to the top with the molten metal, a riser mould is immediately placed on it, and molten slag at 1500—1700° poured in, whereby additional heat is given to the upper part of the cast mass. After solidification the slag is cut away together with the casting.

M. E. NOTTAGE.

Precipitation of metals from solution by the addition of iron. F. A. and G. GRUENNER (G.P. 448,597, 3.1.26).—Porous lumps of coke containing finely-divided iron embedded in them are used, *e.g.*, for precipitating copper from solutions of its compounds.

L. A. COLES.

Leaching of [copper] ores. MINERALS SEPARATION, LTD. From T. J. TAPLIN (B.P. 294,921, 1.4.27).—In the process in which copper silicate ores are heated at 400—500° in a reducing atmosphere and the copper is leached out by an ammoniacal solution, the slimes are first removed from the reduced ore by classification or screening and subjected to oil flotation using coal-tar oil and alkali xanthates, the concentrate being mixed with the coarse material for leaching.

A. R. POWELL.

Copper alloys. C. BILLINGTON (B.P. 295,769, 25.5.27).—Copper alloys containing at least 7% Mn together with Al (2—9%), or Al (2—9%) and Zn (2—8%), or Pb (3—20%) and Zn (2—8%) are described. Up to 1% of chromium, nickel, vanadium, molybdenum, or iron may also be added.

M. E. NOTTAGE.

Alloy. F. VISINTAINER (U.S.P. 1,680,577, 14.8.28. Appl., 2.6.26).—The alloy contains 85—89% Cu, 9—13% Al, 0.25—0.65% Fe, 0.3—0.7% Si, and 0.07—0.12% Sn.

F. G. CROSSE.

Alloys for the manufacture of motor-car brakes etc. A. NEGUI (F.P. 624,257, 1.3.26).—Copper-aluminium alloys containing up to 8% Al are used.

L. A. COLES.

Purification of copper and copper alloys. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of I. R. VALENTINE (B.P. 276,341, 17.8.27. U.S., 18.8.26).—Gases may be removed from castings of copper or copper alloys by heating the metal to about 1200° and adding a deoxidising alloy containing approx. 90% of an alkaline-earth

metal, *e.g.*, magnesium, and 10% of a metal such as aluminium, 1 pt. by wt. of the alloy being added to approx. 320 pts. by wt. of the cupriforous metal.

M. E. NOTTAGE.

Welding of copper and copper alloys. F. M. BECKET, Assr. to ELECTRO METALLURGICAL CO. (U.S.P. 1,680,844, 14.8.28. Appl., 21.2.22).—A welding rod consists of an alloy of copper with 1.25–4% Si.

F. G. CROSSE.

[Copper] welding rod. P. P. PIPES, Assr. to OHIO BRASS CO. (U.S.P. 1,681,904, 21.8.28. Appl., 18.3.27).—A copper rod containing 0.5–1% Sn and 0.1–0.2% P is claimed.

F. G. CROSSE.

Welding rod. N. B. PILLING and J. G. SCHOENER, Assrs. to INTERNAT. NICKEL CO., INC. (U.S.P. 1,679,003, 31.7.28. Appl., 5.12.27).—A welding electrode comprises a nickel rod rendered malleable by the addition of an alkaline-earth metal and coated with a mass containing a titanium alloy and a calcium alloy in such proportions that the titanium in the coating is 0.1–10% and the calcium 0.1–8% of the weight of the electrode.

A. R. POWELL.

Extraction of tin from slime tin, tin-bearing ores, etc. F. L. WILDER, E. MORRIS, E. SCHIFF, and E. S. KING (B.P. 294,703, 6.2.28).—The tin material is passed through an air-tight tubular retort through which sulphur vapours are circulated at 200–300° to convert the tin into sulphide without volatilising more than a small quantity. The product is cooled out of access to the air and leached with a 1% solution of ammonium polysulphide containing 0.5–1% Sn in solution; and the liquor is electrolysed between a tin cathode and a lead-silver anode. The small quantity of tin volatilised in the retort is collected in ammonium carbonate solution, recovered, and added to the main electrolyte.

A. R. POWELL.

Separation of tin from tin-plated scrap etc. L. U. LA CORSA (B.P. 287,892, 23.12.27. Italy, 29.3.27).—Tinplate is treated with a solution of ferric or stannic chloride, and the reduced solution is re-oxidised by treatment with a chlorinating agent and used again, this procedure being repeated until a solution of high tin concentration is obtained. Alternatively, ferric sulphate solution is used for the detinning operation and is regenerated by treatment with sulphur dioxide and oxygen in the presence of a catalyst.

A. R. POWELL.

Light metal [magnesium-tin] alloy. J. A. GANN, Assr. to DOW CHEM. CO. (U.S.P. 1,680,262, 7.8.28. Appl., 27.10.21. Renewed 26.8.26).—The alloy contains 80–99.5% Mg and 20–0.5% Sn.

F. G. CROSSE.

Method and apparatus for condensing zinc vapours. L. MELLERSH-JACKSON. From NEW JERSEY ZINC CO. (B.P. 295,115, 8.7.27).—A zinc condenser comprises a vertical cylinder lined with refractory material and provided with an inverted conical bottom for collecting the molten zinc. The cylindrical part of the condenser is filled with vertical, graphite or carbon tubes, the spaces between which are packed with a paste of graphite or carbon and fireclay. The diameters of the tubes in the middle of the cylinder are smaller than those of the tubes round the walls. The temperature at the top of the tubes is maintained about 200–300°

lower than that at the bottom, which is so adjusted that the accumulated zinc in the conical sump still evolves an appreciable quantity of vapour to enrich the incoming gases. These deposit their zinc content as they rise through the tubes in such a way that the condensed metal runs down the walls as a thin film which assists in further condensation of zinc without the formation of blue powder. A recovery of 95% of the total zinc in the molten form is claimed.

A. R. POWELL.

Method of pulverising and alloying nickel. W. R. VEAZEY, Assr. to DOW CHEM. CO. (U.S.P. 1,680,825, 14.8.28. Appl., 9.8.23).—Nickel and magnesium are heated together at a temperature between their respective m.p. until sufficient magnesium is absorbed by the nickel to render it readily pulverisable.

F. G. CROSSE.

Aluminium alloy of high resistance. H. BÉNIT (U.S.P. 1,680,004, 7.8.28. Appl., 25.2.26. Ger., 19.2.25).—A light alloy, capable of being repeatedly melted without losing its characteristic properties, is composed of commercial aluminium (containing 0.5–2% of impurities) alloyed with 2% Cu, 0.37% Ni, 0.27% W, and 0.45% Mg.

F. G. CROSSE.

Age-hardening process [for lead]. R. S. DEAN and W. E. HUDSON, Assrs. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,675,644, 3.7.28. Appl., 16.9.24. Renewed 20.10.27).—An age-hardenable lead alloy is heated a number of times at the temperature at which the maximum amount of alloying element goes into solid solution, subjected to work between successive heatings, and finally quenched and aged.

A. R. POWELL.

Treatment of lead[antimony] alloys. [A–E] R. S. DEAN and W. E. HUDSON, and [F] R. S. DEAN, Assrs. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,674,954—9, 26.6.28. Appl., [A–D] 5.2.24, [E] 21.6.24, [F] 23.1.26).—(A) Lead alloyed with a small quantity of antimony is heat-treated to form a supersaturated solid solution which is further treated to obtain a more stable alloy. (B) Lead-antimony alloys for extrusion are preheated for 72 hrs. at 240°. (C) An alloy of lead with up to 2.5% Sb is heated just below its m.p. to obtain a homogeneous solid solution which is immediately extruded into cable sheath, quenched, and aged. (D) Articles of antimonial lead are heat-treated to obtain a solid solution, quenched below 100°, and subsequently aged below 100°. (E) A lead-antimony alloy in which the antimony particles are uniformly distributed in a state of fine subdivision throughout the lead is claimed. (F) A process for controlling the mechanical properties of a hardenable lead alloy comprises heating the solid alloy for such a time and at such a temperature that a supersaturated solid solution is obtained at a controllable rate.

A. R. POWELL.

Production of moulded articles from molybdenum and its alloys. P. A. KURT (Austr.P. 106,973, 12.3.25).—Pure molybdenum obtained by heating the oxide in a current of hydrogen, alone or mixed with one or more metals of the iron group or with silicon, preferably with the addition of carbon, titanium, zirconium, or boron, is fused by electrical

resistance and shaped in moulds. The products are resistant to attack by acids, especially hydrochloric acid. L. A. COLES.

Apparatus for the decomposition of alkali and alkaline-earth amalgams. I. G. FARBERIND. A.-G., Assees. of E. HEINZE (G.P. 448,892, 2.4.25).—The stirring device in the apparatus described in G.P. 427,236 and 456,290 is constructed of charcoal, coke, graphite, etc., or of superposed sections of these, alone or in combination with alloys of iron, chromium, or nickel. L. A. COLES.

Coating for protecting the inner surfaces of hollow bodies, such as tubes etc. BRIT. MANNESEMANN TUBE CO., LTD., and R. F. WEISER (B.P. 295,267, 5.4.27).—An internal lining for tubes consists of native Trinidad bitumen (including its natural mineral content of about 40% of silicious material) mixed with 20% of asphaltic mineral oil. A. R. POWELL.

Metal plating. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of R. PALMER (B.P. 284,302, 24.1.28. U.S., 27.1.27).—Metal articles may be coated with a protective covering of a metal soluble in the base metal by applying a first coating of a metal, such as silver or copper, which is relatively insoluble in the base metal, superimposing the more soluble protective metal, and subsequently heating in a reducing atmosphere at slightly below the m.p. of the first coating. M. E. NOTTAGE.

Cadmium plating. L. R. WESTBROOK, Assr. to GRASSELLI CHEM. CO. (U.S.P. 1,681,509, 21.8.28. Appl., 23.11.26).—The plating solution is comprised of a mixture of 50 pts. of cadmium hydroxide, 0.18 pt. of nickel as a compound which is soluble in an aqueous solution of alkali cyanide, about 38 pts. of sodium sulphate, and 11.5 pts. of galac. F. G. CROSSE.

Open-hearth furnace. F. B. MCKUNE, Assr. to OPEN HEARTH COMBUSTION CO. (Re-issue 17,071, 28.8.28, of U.S.P. 1,339,855, 11.5.20).—See B., 1928, 128.

Apparatus for refining metals. J. MAXIMOFF, M. S. DE COSTA, and R. P. D. KREBS (U.S.P. 1,681,191, 21.8.28. Appl., 6.1.26. Fr., 28.11.25).—See B.P. 262,136; B., 1927, 912.

Production of volatile metals from their sulphides. H. G. FLODIN and E. G. T. GUSTAFSSON, Assrs. to H. BROADBY (U.S.P. 1,680,098, 7.8.28. Appl., 10.4.24).—See B.P. 215,004; B., 1925, 362.

Alloy [for jets used in spinning artificial silk]. W. P. DREAPER (U.S.P. 1,680,598, 14.8.28. Appl., 30.7.26. U.K., 5.8.25).—See B.P. 260,672; B., 1927, 47.

Lead alloy. H. YOSHIKAWA (U.S.P. 1,681,272, 21.8.28. Appl., 12.11.26. Japan, 30.11.25).—See B.P. 253,920; B., 1927, 338.

Apparatus for producing [metallic] sheets and similar articles electrolytically. J. C. F. A. SCHÜTTE (U.S.P. 1,682,426, 28.8.28. Appl., 6.10.27. Ger., 29.12.25).—See B.P. 292,015; B., 1928, 610.

Moulding briquettes of iron ore or iron-bearing materials, and apparatus therefor. K. and E. GRÖPPEL and A. WASCHKAU (MASCHINENFABR. FR. GRÖPPEL, C. LÜHRIG'S NACHFOLGER), and C. P. DEBUCH (B.P. 295,910, 2.3.28).

Manufacture of [dental] amalgams. DEUTS.

GOLD- U. SILBER-SCHNEIDANSTALT, VORM. ROESSLER, and F. SCHOENBECK (B.P. 295,163, 29.11.27).

Covering and protecting the surfaces of hollow bodies [with bitumen etc.]. BRIT. MANNESEMANN TUBE CO., LTD., and R. F. WEISER (B.P. 295,340, 5.4.27).

[Wire-reinforced fabric] binder for covering the surfaces of pipes and tubes. BRIT. MANNESEMANN TUBE CO., LTD., and W. KOEHN (B.P. 295,297, 5.4.27).

Coating wires. INTERNAT. GEN. ELECTRIC CO., INC., Assees. of ALLGEM. ELEKTRIZITÄTS-GES. (B.P. 290,252, 10.5.28. Ger., 11.5.27).

Apparatus for electromagnetic separation of ores. A. DAVIES (B.P. 295,417, 5.5. and 7.11.27).

Washing of minerals (B.P. 285,864 and 286,281).—See II. **Electrodes** (B.P. 295,514).—See XI.

XI.—ELECTROTECHNICS.

High-frequency [induction] furnace with rotating spark-gap and variable frequency. M. H. KRAEMER (Stahl u. Eisen, 1928, 48, 1120—1124).—In a new type of high-frequency induction furnace the rotating spark-gap is obtained by means of a motor-driven steel rotor carrying spaced copper electrodes working between two stationary water-cooled copper electrodes with a gap of 0.75 mm. The current is transformed up from 220 volts to 7000—8000 volts in the usual oil-transformer, then passes through a battery of ten condensers, each of 0.066 microfarad in parallel, across the spark-gap to the self-induction coil of the furnace. With suitable sizes and windings of the latter any charge from 100 g. to 5 kg. or more of iron can be melted in less than 20 min., and a frequency of 14,000—230,000 cycles/sec. can be obtained. A. R. POWELL.

Nickel anodes and their use. W. S. BARROWS (Can. Foundry, 1926, 17, 14—16).—It is easier to maintain an equilibrium between anode and cathode efficiency with well cast nickel anodes of 90—92% purity than with poorly cast anodes of 95—97% purity. Sulphur and copper are injurious. Red stains when the bath is idle may be due to cobalt. Tin makes casting easier, and like silicon it increases the formation and maintenance of slime on the anodes. Iron and manganese are generally harmful. CHEMICAL ABSTRACTS.

Conductivity of insulating oils. BLACK.—See II. **Electro-osmose tanning.** POLLAK.—See XV.

PATENTS.

Induction furnace. P. H. BRACE, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,680,407, 14.8.28. Appl., 29.8.27).—Steam issuing from a water-cooled inductor coil is used to generate electrical energy which passes to the electrical circuit of the coil.

J. S. G. THOMAS.

Manufacture of electric cables etc. A./S. DE NORSKE SALTVERKER (F.P. 622,560, 5.10.26. Norw., 9.10.25).—A metallic conductor, e.g., copper, is coated with magnesium and enclosed in a tube through which superheated steam or water under pressure is passed at 450°, so that the magnesium is converted into its hydroxide, which fills the space between the conductor and the tube. The hydroxide may be converted into the oxide by stronger heating. L. A. COLES.

Manufacture of electrodes, welding rods, etc. for use in welding and depositing metal. B. TURNER and FERRO-ARC WELDING CO., LTD. (B.P. 295,514, 26.8.27).—In a twin electrode for welding on a polyphase circuit, an insulating material, *e.g.*, white asbestos, having a higher m.p. than that of the material, *e.g.*, blue asbestos, binding the wires outside, is inserted between the wires. J. S. G. THOMAS.

Manufacture of high resistances. T. D. PARKIN (B.P. 295,444, 13.5.27).—A film or thin layer of finely-divided conducting material, *e.g.*, platinum, silver, "dag," &c., is printed, under pressure, upon a moulding of insulating material, *e.g.*, a phenol-formaldehyde condensation product. J. S. G. THOMAS.

Magnetic material [copper-nickel alloy]. G. W. ELMEN, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,675,884, 3.7.28. Appl., 29.5.26).—A magnetic copper-nickel alloy having a higher permeability than iron in weak magnetic fields is claimed. A. R. POWELL.

Thermionic valve. C. TIETIG, Assr. to A. ANDRIESSEN (U.S.P. 1,673,850, 19.6.28. Appl., 1.5.22).—The electron stream is produced by the continuous combustion of a mixture of a metal and a metallic oxide (the latter supplying all the necessary oxygen) packed in a thin metal cylinder. F. G. CLARKE.

Argon-mercury discharge tube. Luminous tube. R. R. MACHLETT, Assr. to RAINBOW LIGHT, INC. (U.S.P. 1,680,271—2, 7.8.28. Appl., [A] 9.7.27, [B] 17.11.27).—(A) A luminous tube contains argon and mercury vapour, the latter being uniformly evolved from the glass walls of the tube. (B) A transparent tubular vessel filled with a rare gas of the atmosphere under reduced pressure is provided with interior electrodes of absorbent conducting material containing occluded alkali-metal vapour. J. S. G. THOMAS.

Dispersing magnesium in vacuum tubes. S. LOEWE (B.P. 279,844, 24.10.27. Ger., 26.10.26).—Magnesium, to be dispersed, is movably mounted within the vacuum tube, and, after discharge of gas from the elements of the tube is complete, is brought into immediate contact with the hot anode. J. S. G. THOMAS.

Coating for glass surfaces [of incandescence lamps]. H. D. BLAKE and J. E. STONE, Assrs. to GEN. ELECTRIC CO. (U.S.P. 1,672,857, 5.6.28. Appl., 27.2.22).—Incandescence lamps are coated with a mixture of china clay and sodium silicate solution and then dipped in a fixing bath comprising a solution of sodium hydrogen fluoride or of sodium or ammonium chlorides and sulphuric acid. A. R. POWELL.

Galvanic battery. A. M. CODD (B.P. 295,743, 18.5.27).—A zinc electrode situated at the bottom of the cell is protected by a porous diaphragm, *e.g.*, of kieselguhr, sand, glass, etc., and immersed in an electrolyte consisting of a solution of ferric chloride and containing a carbon electrode. J. S. G. THOMAS.

Storage-battery grid. R. S. DEAN and W. E. HUDSON, Assrs. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,675,643, 3.7.28. Appl., 5.2.24. Renewed 27.9.27).—The grid consists of a lead alloy in which the alloying element is present in the form of finely-divided particles dispersed uniformly throughout the mass. A. R. POWELL.

Manufacture of accumulator plates. I. G. FARBENIND. A.-G. (B.P. 285,354, 13.2.28. Ger., 12.2.27. Addn. to B.P. 284,352; B., 1928, 717).—The lead skeleton previously described is provided with a coating, *e.g.*, of oxide, peroxide, sulphide, or sulphate of lead, which is a good thermal insulator and easily removable before or during formation of the electrodes. J. S. G. THOMAS.

Electrolyte. H. O. SIEGMUND, Assr. to WESTERN ELECTRIC CO., INC. (U.S.P. 1,675,828, 3.7.28. Appl., 29.12.24).—A film-forming electrolyte comprises a mixture of ammonia and boric acid solutions in such proportions that the resulting solution has p_H 6.8—7.8 and a specific electrical resistance of 150—800 ohms/cm.³ A. R. POWELL.

Rectifier electrolyte. E. W. ENGLE, Assr. to FANSTEEL PRODUCTS CO., INC. (U.S.P. 1,680,210, 7.8.28. Appl., 7.1.27).—In an electrolytic rectifier having tantalum and lead electrodes, a small proportion of a cobalt salt, *e.g.*, cobalt sulphate, is dissolved in the dilute sulphuric acid electrolyte in order to prevent destruction of the lead electrode. J. S. G. THOMAS.

[Multi-layer] insulating material. A. JOFFÉ (B.P. 286,320, 8.9.26).

Mounting or suspension of resistances in electric furnaces. H. WIGGIN & CO., LTD., and A. G. LOBLEY (B.P. 295,299, 8.4.27).

Perforated container for rotating-barrel electroplating apparatus. W. CANNING & CO., LTD., and G. A. POPE (B.P. 295,791, 18.6.27).

Galvanic batteries. OLDHAM & SON, LTD., and W. D. WILDE (B.P. 295,516, 1.9. and 13.9.27).

[Forming hollow, cylindrical elements for] primary cells, and secondary cells. J. S. CROSSLEY (B.P. 295,219 and 295,220, 4.4.27).

Accumulator contact plates. CONCORDIA ELEKTRIZITÄTS A.-G., and W. GOSMANN (B.P. 295,561, 19.3.28).

Gas-filled electric discharge tube. H. WADE. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 295,293, 2.3.27).

Electric discharge [space current] device. BRIT. THOMSON-HOUSTON CO., LTD., Asses. of R. B. PRINDLE (B.P. 267,545, 11.3.27. U.S., 12.3.26).

Coloured or diffusing coating for [bulbs of] incandescence lamps etc. BRIT. THOMSON-HOUSTON CO., LTD., Asses. of M. PIPKIN (B.P. 277,695, 19.9.27. U.S., 20.9.26).

Electrical precipitation (B.P. 295,890).—See I. **Heating carbonaceous materials** (B.P. 279,825). **Reducing gases** (B.P. 294,838). **Treatment of hydrocarbons** (B.P. 294,494).—See II. **Radioactive substances** (B.P. 257,957). **Hydrogen peroxide etc.** (B.P. 295,137).—See VII. **Fused cement** (Addn. F.P. 31,931).—See IX. **Magnetic alloys** (B.P. 295,443). **Metals from sulphides** (B.P. 280,540). **Welding rod** (U.S.P. 1,679,003). **Extraction of tin** (B.P. 294,703). **Cadmium plating** (U.S.P. 1,681,509).—See X.

XII.—FATS; OILS; WAXES.

Defects in methods of determining unsaponified neutral fat in soaps. H. KRÖPER (Chem. Umschau, 1928, 35, 213—217).—When ethyl ether is used for a Soxhlet extraction of soap, considerable quantities of fatty acids are liberated for which correction must be made after removal of the dissolved soap and glycerin. Approximately correct values are obtained by the use of light petroleum, but it is necessary previously to dry the soap, which involves a risk of further saponification due to the presence of neutral fat and free alkali. The best results are realised by neutralisation of the "total fatty matter" by excess alkali in 50% alcohol and wet extraction with light petroleum; the extract must be washed with alkaline 50% alcohol. The residue obtained by evaporation of the solvent is weighed, neutralised if necessary, and the ester value determined, from which the proportion of unsaponified neutral fat in the soap is calculated. E. LEWKOWITSCH.

Simplified determination of cresol in cresol soap preparations. G. KOGAN (Pharm. Zentr., 1928, 69, 536—537).—The alkali salts of the fatty acids are converted into the barium salts in presence of ether, which dissolves the cresols and hydrocarbons. If the proportion of hydrocarbons present is sufficient to make the aqueous liquor milky, the ethereal solution after separation of the barium salts of the fatty acids is shaken with sodium hydroxide solution, and the cresols so removed are precipitated by acid, extracted with ether, and dried and weighed in the usual manner. S. I. LEVY.

Possibility of different applications of linseed oil conditioned by geographical origin. Quantitative analysis and paint technology of a Calcutta oil. A. EIBNER and F. BROSEL (Chem. Umschau, 1928, 35, 157—166).—The work of Ivanov (A., 1927, 906) on the influence of climate on the fatty acid content of linseed and conifer seed oils is reviewed. A Calcutta linseed oil had d_{20}^{20} 0.9273, η (Engler) 8.25/18.9° (5.06/31.4°), n_D^{20} 1.47509, iodine value 181.7, acid value 0.90, saponification value 194.4, mean mol. wt. of fatty acids 273.3, unsaponifiable matter 0.96%. From the bromide values the following percentage fatty acid contents of the Dutch and Calcutta linseed oils were respectively derived: Total unsaturated acids 86.1, 85.1; α -linolenic acid 20.1, 22.93; α -linoleic acid 17.0, 17.43; β -linolenic acid 2.7, 22.82; β -linoleic acid 41.8, 4.32; oleic acid 4.5, 17.61. By bromination of the oil itself the bromides of di- α -linolenic- α -linoleic glyceride and of di- α -linolenic-oleic glyceride were isolated, together with a new *bromoglyceride*, m.p. 143—144°, isomeric with the first-mentioned (probably bromodi- α -linolenic- β -linoleic glyceride), and the approximate glyceride composition of the oil calculated. In technological tests the Calcutta oil behaved normally, the exceptional amount of oleic acid being compensated by the decreased amount of linoleic acid, and balancing the additional drying activity to be expected from the higher β -linolenic acid content. It is emphasised that the iodine value alone is not a criterion of a good technical linseed oil, since it is affected by other unsaturated acids than the α -linolenic acid, which is principally responsible for the drying qualities of the oil. E. LEWKOWITSCH.

Determination of colour number of oils, lacquers, and similar products. E. STOCK (Farben-Ztg., 1928, 33, 2967—2969).—An improved tintometer ("Komparator nach Hellige-Stock") is proposed which is simple, rapid in use, and gives accurate reproducible results; it is suitable for quantitative colorimetric analysis and for the determination of p_H by the Walpole method.

E. LEWKOWITSCH.

Oleomargarine. KERR.—See XIX.

PATENTS.

Extraction of fats from vegetable matter [cacao beans]. I. S. HOCKER, Assr. to HOCKER CORP. (U.S.P. 1,680,943, 14.8.28. Appl., 26.1.24).—A suspension in water of the finely-divided beans is heated to dissolve the soluble constituents and gelatinise the starch. The latter is then converted into sugar and the fat liberated by treatment with a diastatic ferment. Coalescence of the fat globules is effected during the subsequent alcoholic fermentation, due to the evolution of gas. F. G. CLARKE.

Distillation of solvents from solutions and deodorisation of oils, fats, etc. P. L. FAUTH (B.P. 285,380, 13.2.28. Ger., 14.2.27).—The solutions are sprayed into a distillation chamber maintained by external steam-heating at 100—120°. The vapour of the solvent is withdrawn at the top of the chamber, and the oil collecting at the bottom is passed through one or more deodorising vessels in which it is introduced as a spray at the top and is treated with a counter-current of superheated steam, the vapour being withdrawn at the top under vacuum through a regulating device which maintains the contents slightly above atmospheric pressure. L. A. COLES.

Cooling device for fatty emulsions or the like. W. G. SCHRÖDER (U.S.P. 1,679,685, 7.8.28. Appl., 20.10.26. Ger., 20.10.25).—See B.P. 269,416; B., 1927, 531.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Absorption of basic dyes by complex mineral salts. M. MICHELS (Bull. Soc. Ind. Mulhouse, 1928, 94, 344—357).—Chromic acid and water-soluble chromates yield precipitates with basic dyes which are slightly soluble in water and benzene; the precipitates with rhodamine dyes crystallise readily. Alkali cyanides and thiocyanates precipitate basic dyes, the precipitates with cyanides being more water-soluble than those with thiocyanates. Cyanides, chromates, sulphites, etc. which are insoluble in water do not absorb basic dyes unless previously treated so that they are partially converted into complex salts. *E.g.*, zinc sulphite absorbs basic dyes at 100°, but not in the cold, and zinc cyanide only absorbs basic dyes after heating with an alkali (partial formation of zincocyanate). Complex substances derived from cyanides and thiocyanates absorb basic dyes, the products being usually slightly soluble or insoluble in water and readily obtained crystalline. Basic dyes are absorbed by the complex cyanides and cyanates of silver, mercury, copper, zinc, iron, nickel, and cobalt, and if they are extremely finely divided or capable of forming aqueous colloidal solutions the products are fast to light and washing with water. Prussian blue does not absorb basic dyes. The absorption

compounds of basic dyes with nickel and cobalt ferrocyanides are unstable, particularly the cobalt compounds. Both ferri- and ferro-cyanides precipitate basic dyes, the products obtained with the latter being less water-soluble than those obtained with ferricyanides. Zinc ferrocyanide absorbs basic dyes strongly, the fastness of the products being proportional to the fineness of division of the absorbent particles. Cupric ferrocyanide commences to absorb basic dyes when heated in water at 50–100°, or in the cold after treatment with a cupric salt; the products are fast to light but not to washing. Cuprous ferrocyanide absorbs basic dyes strongly, the products being very fast to light and washing; it is also a better mordant than tannic acid for basic dyes on cotton. Silicic acid absorbs basic dyes, but the products are not fast to light or washing unless the colloidal acid is used; the latter also absorbs di- but not tri-phenylmethane dyes, the last-named being precipitated by molybdic and tungstic acids. Molybdic acid, being more colloidal than tungstic acid, absorbs basic dyes more strongly. Uranium trioxide, colloidal sulphur, and zinc sulphide absorb basic dyes feebly.

A. J. HALL.

Applicability of the analytical quartz lamp. HEIN and RETTER.—See I. **Linseed oils.** EIBNER and BROSEL. **Colour number of lacquers.** STOCK.—See XII.

PATENTS.

Production of cellulose ester resin lacquers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 294,911, 29.4.27).—Hard resins, *e.g.*, copals, may be incorporated into cellulose ester lacquers in the presence of suitable softening agents, *e.g.*, tricresyl phosphate.

S. S. WOOLF.

Nitrocellulose lacquer composition. C. L. GABRIEL and C. BOGIN, Assrs. to COMMERCIAL SOLVENTS CORP. (U.S.P. 1,763,111, 12.6.28. Appl., 21.5.25).—The use of *n*-butyl lactate (with ethyl acetate and benzene, toluene, and xylene) lacquers containing nitrocellulose and gums results in slow, even drying and smooth films.

C. HOLLINS.

Manufacture of coloured coating lacquers. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 294,912, 29.4.27).—Coloured compositions (*cf.* B.P. 247,238 and 293,485; B., 1926, 315; 1928, 668) are dissolved in mixtures containing butyl or amyl esters, aromatic hydrocarbons or their halogen compounds boiling above 100°, and, preferably, aliphatic alcohols containing at least four carbon atoms per mol. The resulting lacquers dry with high gloss.

S. S. WOOLF.

Production of high-melting coumarone resins. I. G. FARBENIND. A.-G. (G.P. 446,707, 11.1.18).—Low-melting coal-tar distillates are stirred with small quantities of hydrated metal salts with a polymerising action, *e.g.*, aluminium chloride or ferric chloride, without external heating.

L. A. COLES.

Manufacture of condensation products of urea and formaldehyde. I. G. FARBENIND. A.-G. (B.P. 266,752, 28.2.27. Ger., 27.2.26; *cf.* B.P. 261,029; B., 1928, 532).—Dimethylolurea or a derived product is condensed under mild conditions with acid condensing agents in alcohols, alone or mixed with other organic

solvents. The condensation is stopped before high-molecular colloidal compounds are formed.

S. S. WOOLF.

Condensation products from dihydroxydiphenylethane and their manufacture. E. C. R. MARKS. From BAKELITE CORP. (B.P. 295,442, 13.5.27).—Resins for varnishes and for moulding-powders are made by heating 4:4'-dihydroxydiphenylethane with formaldehyde, hexamethylenetetramine, etc. at 180–200°.

C. HOLLINS.

Treating solutions [for recovering gummy or plastic substances]. W. S. JOHNSTON and A. W. KEEN, Assrs. to NAUGATUCK CHEM. Co. (U.S.P. 1,673,685, 12.6.28. Appl., 15.6.27).—The solution, *e.g.*, of polymerised styrol in ethylbenzene, is mixed under pressure with a hot gas, *e.g.*, steam at 115°, and the mixture is expanded through a nozzle whereby the ethylbenzene is steam-distilled leaving a residue of the plastic polymerised styrol.

A. R. POWELL.

Manufacture of composite titanium pigment. L. E. BARTON and L. W. RYAN, Assrs. to TITANIUM PIGMENT Co., Inc. (U.S.P. 1,680,316, 14.8.28. Appl., 8.7.25).—See B.P. 281,459; B., 1928, 131.

Manufacture of a water-soluble [resinous] condensation product. K. DAIMLER, F. MARSHALL, and G. BALLE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,635,883, 12.7.27. Appl., 10.12.24. Ger., 12.8.22).—See B.P. 240,318; B., 1925, 965.

Purifying phenol-aldehyde resins. F. SEEBACH, Assr. to BAKELITE GES.M.B.H. (U.S.P. 1,681,368—9, 21.8.28. Appl., 21.1.26. Ger., 27.1.25).—See B.P. 246,834; B., 1927, 684.

Nitrocellulose (B.P. 280,922).—See V. **Zinc pigments** (B.P. 282,706).—See VII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

PATENTS.

Treatment of rubber, and products obtained thereby. S. M. CADWELL, Assr. to NAUGATUCK CHEM. Co. (U.S.P. 1,673,550, 12.6.28. Appl., 17.3.27).—The products obtained by the action of *p*-toluenesulphonyl chloride or of sulphuryl chloride upon dialkylthiocarbamates are vulcanisation accelerators, the effect being said to be 30–40% greater than that of the thiocarbamates. All products of the type R·S·SO₂·R' are claimed.

C. HOLLINS.

Treatment of rubber with aldehyde-amine condensation products. S. M. CADWELL, Assr. to NAUGATUCK CHEM. Co. (U.S.P. 1,673,549, 12.6.28. Appl., 7.1.27).—The hexahydro-1:3:5-triazines obtainable from formaldehyde and aliphatic amines (specially ethylamine) are both accelerators and anti-agers. To obtain the anti-ageing effect, excess of the agent is used over that required for vulcanisation, over-vulcanisation being avoided by the use of suitably small proportion of sulphur; *e.g.*, to 100 pts. of rubber are added 5 pts. of zinc oxide, 2.6 pts. of sulphur, and 5–10 pts. of 1:3:5-triethylhexahydro-1:3:5-triazine.

C. HOLLINS.

Manufacture of material resembling rubber. C. A. HOUQUES-FOURCADE (F.P. 622,896, 9.10.26).—Marine algae are heated with 5–10% of sodium car-

bonate solution, and the product, after removal of insoluble residues, is heated with sulphur to about 110°, or is treated with a solution of sulphur chloride in carbon disulphide or carbon tetrachloride, after treatment, if necessary, with acetic acid, alum, or other coagulants.

L. A. COLES.

Vulcanisation of rubber. L. B. SEBRELL, Asst. to GOODYEAR TIRE & RUBBER Co. (U.S.P. 1,679,548, 7.8.28. Appl., 12.2.26).—See B.P. 265,920; B., 1928, 377.

Manufacture of substituted thiouram polysulphides. G. S. WHITEBY, Asst. to ROESSLER & HASSLACHER CHEM. Co. (U.S.P. 1,681,717, 21.8.28. Appl., 27.1.26).—See B.P. 265,169; B., 1928, 203.

Carbon black (U.S.P. 1,673,496).—See II.

XV.—LEATHER; GLUE.

Problems of fat-liquors. W. SCHINDLER (Collegium, 1928, 241—274).—Fat-liquors consist of small droplets, which, being electrically charged, repel each other and so prevent coalescence. They gradually lose their charge and then coalesce. The stability of the emulsion depends on the conductivity, the size of the drops, the surface tension of the oil/aqueous dispersion medium, the difference in the sp. gr. of the oil and the medium, the viscosity of the latter, and the surface film which surrounds the oil drops. "Structural viscosity" has been observed in some aqueous emulsions. The Brownian movement does not occur much in leather-trade emulsions. Fat-liquors contain three phases, and give rise to four kinds of viscosity, viz.: (a) of the emulsion itself, (b) of the disperse-phase oil, (c) of the surface film surrounding the oil drops, and (d) of the dispersive medium. The structural viscosity of different sulphonated oils tested was low at p_H 7.5—9.5, above 9.5 it increased and then diminished again. Ammonia-neutralised sulphonated oils showed a much greater structural viscosity than those neutralised with soda. Different sulphonated oils (neatsfoot, castor, cod) have been fractionated into five different products: α_1 , α_2 , β_1 , β_2 , and γ . Of these, α_1 and γ , being neutral, do not emulsify but have good fat-liquoring properties owing to their high viscosity; α_2 consists chiefly of free and oxidised fatty acids of low viscosity, hence do not fat-liquor well; β comprises oxidised fats, and the emulsent properties of sulphonated oils can be judged from the amount of β and its sulphur trioxide content. The speed of fat absorption by leather is greatest at the commencement of the fat-liquoring. Soap solutions and soap-mineral oil emulsions are absorbed more slowly and incompletely than mixtures containing sulphonated oils. Additions of mineral oil or untreated fatty oil increase the speed of absorption but not the total amount absorbed. Sulphonated oils are absorbed more quickly and in greater quantity than is neutral oil from emulsions. Every emulsent has its own optimum p_H value, which is higher for soaps than for sulphonated oils. Increase in p_H diminishes the size of the oil drops, and the fat absorption-temperature curve is thereby flattened. Increase in temperature causes increased fat absorption but diminished stability.

D. WOODROFFE.

[Sodium] sulphide stains on white hide. G. W.

SCHULTZ (J. Amer. Leather Chem. Assoc., 1928, 23, 356—361).—Frigorifico hides rarely show these stains. Hides limed for 48 hrs. in a lime liquor without sulphide and then put into a sulphide-lime solution show no stains. Soaking in an alkaline liquor (0.75% sodium hydroxide) prevented sulphide stains. The stains are not caused by the presence of iron. They are more prevalent on badly cured hides (especially green-salted domestic hides), and increase in intensity and extent as hides are kept in the cured state; during such increase the evidence of salt stains increases also. It is concluded that the blue stain with sulphide is probably caused by some decomposition product of the hide.

D. WOODROFFE.

Tanning of marine animal skins. C. ZIEGLER (Cuir techn., 1927, 19, 2—7, 28—29; Chem. Zentr., 1927, II, 1921—1922).—The raw skins are preserved by salting repeatedly, or by immersion in brine for 8 days. Before tanning the skins are soaked in a 0.2—1% solution of sodium hydroxide and then treated with hydrochloric acid to remove the scales. Pancreatic preparations are used for bating the skins, after which for chrome tannage they are treated with ammonium chloride and tanned by the two-bath chrome process. Acid liquors must be avoided in the vegetable tanning process as the skins are very susceptible to acids. Chestnut, quebracho, gambier, and oakbark are used on them. Only small skins are chamoised.

D. WOODROFFE.

Electro-osmose tanning. L. POILAK (Gerber, 1926, 52, 174—175, 205—206; Chem. Zentr., 1927, II, 1922).—Colloids are electrically charged and are precipitated by oppositely charged colloids, electrodes, or diaphragms. In electro-osmose tanning the hides are arranged in the liquors between two electrodes separated by diaphragms. The current of electricity charges the pelts positively and renders them better capable of absorbing the negatively charged tannin. About 0.1—0.125 kw.-hrs. at 100 volts (max.) is necessary for 1 kg. of sole leather. Preliminary tannage is done in normal liquors without electricity, and the tannage is completed in drums. Osmotic tannage curtails the time of tanning and increases the yield of leather.

D. WOODROFFE.

Tannin content of Alaskan Mountain hemlock bark (*Tsuga mertensiana*). P. B. DAVIDSON and E. C. SHERRARD (J. Amer. Leather Chem. Assoc., 1928, 23, 371—372).—The average of eight analyses of the oven-dry bark was: tans 12.21%, non-tans 9.18%, insolubles 5.42%.

D. WOODROFFE.

PATENTS.

Tanning materials. R. ALCALAI (B.P. 281,292, 23.11.27. Belg., 23.11.26).—An aqueous solution of a suitable vegetable tanning material containing 9—13% of tannin is mixed with a solution of a normal chromium salt and an aluminium salt, insufficient to cause tanning itself. Neutral salts, e.g., sodium chloride and/or sulphate, may be added to the mixture. The mixture may result in a precipitate which is then separated and used for tanning with or without a vegetable tanning material.

D. WOODROFFE.

Composition for treating hides and skins. H. DODGE (U.S.P. 1,680,136, 7.8.28. Appl., 18.1.28).—A

mixture of formaldehyde, nitre, sodium bicarbonate, and sodium sulphate is claimed. F. G. CROSSE.

Production of white parchment. F. ZNIDARIC (Austr. P. 106,854, 19.1.26).—Depilated hides are treated for about $1\frac{1}{2}$ hrs. with a solution containing arsenic trisulphide and sodium chloride, then with an aqueous solution prepared from anhydrous alum and dilute sulphuric acid or hydrochloric acid, and are finally stretched and dried. L. A. COLES.

Treatment of soya beans. O. JOHNSON, Assr. to I. F. LAUCKS, INC. (U.S.P. 1,680,264, 7.8.28. Appl., 27.5.24).—Juice is extracted from the mass obtained by soaking the beans in water at not above 71° , grinding the moist beans, and adding water at 71° or less and an alkali. A base for an adhesive is obtained by separating the oil from the juice, adding a curdling agent, and removing moisture from the curd. J. S. G. THOMAS.

Protein composition. M. LEVIN (U.S.P. 1,675,181, 26.6.28. Appl., 15.9.27).—An adhesive binder containing extending agents to prevent deterioration and hardening is made by mixing glue, glycerol, water, and hydrol (the mother-liquor of d 1.39—1.54 resulting from the production of sugar by the hydrolysis of starch).

F. R. ENNOS.

Manufacture of condensation products containing sulphur [tanning agents]. O. SPENGLER and A. THURM, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,682,434, 28.8.28. Appl., 3.5.26. Ger., 28.5.25).—See B.P. 252,694; B., 1927, 373.

XVI.—AGRICULTURE.

Significance of hydrogen-ion concentration for the cycle of nitrogen transformation in the soil. C. OLSEN (Compt. rend. Trav. Lab. Carlsberg, 1928, 17, No. 8, 21 pp.).—Culture experiments show that ammonifying organisms are active within the range p_H 3.7—9.0 with an optimum value between p_H 7.0 and 8.5. In acid soils nitrification begins at p_H 3.7, increasing in activity to the maximum at value p_H 8.3 and declining again to the upper limiting value p_H 8.8. In general, between p_H 4.0 and 8.0 the rate of nitrification is greater than the rate of ammonification, and the latter process controls the former. Nitrifying organisms appear to exist in very acid soils, although remaining inactive until the p_H value is adjusted. In some cases re-inoculation is necessary. Air-drying of soils does not exterminate nitrifying organisms, which may therefore be distributed in dust from one locality to another.

A. G. POLLARD.

Effects of limestone and hydrated lime on biochemical activities in acid soils. H. DORSEY (Connecticut Storrs Sta. Bull., 1926, 141, 113—163).—Ground limestone gradually reduces the acidity of an acid soil for 6—8 weeks; hydrated lime acts rapidly, the acidity rising after 2—3 weeks for 8 weeks. Untreated soil shows fluctuations in acidity which are reflected in treated soil. Jones' test is preferred, and accords with the hydrogen-ion test. The ammonifying and nitrifying power of acid soils is increased by limestone.

CHEMICAL ABSTRACTS.

Action of alkali. Soil moisture. M. D. THOMAS and D. S. JENNINGS (Utah Agric. Exp. Sta. Bull., 1926,

No. 198, 65—68).—On addition of sodium salts to soil the sodium replaces calcium, magnesium, and potassium in the silicate complex, yielding a soil partly impermeable to water, and alkaline in reaction. In wet clay soils treated with sodium salts considerable deflocculation was induced.

CHEMICAL ABSTRACTS.

Effects of nitrates on the composition of the potato. W. P. HEADDEN (Colo. Agric. Exp. Sta. Bull., 1928, No. 325, 1—96).—Excess of nitrate is toxic to potatoes, decreasing the yield and cooking quality; the phosphate is decreased and the potash increased.

CHEMICAL ABSTRACTS.

Transformation of nitrogen in rice soil. G. JANSSEN and W. H. METZGER (J. Amer. Soc. Agron., 1928, 20, 459—476).—The nitrate, nitrite, and ammonia in flooded and unflooded soils under greenhouse conditions, treated with green manure, ammonium sulphate, or sodium nitrate, and untreated soils, were determined. The nitrite content was never significant. Half the soils were uncropped, and the others cropped to rice.

CHEMICAL ABSTRACTS.

Soil reaction and nitrogen fixation. P. C. KRISHNA (J. Amer. Soc. Agron., 1928, 20, 515—518).—The amounts of nitrogen fixed increase progressively with decreasing acidity of soils, e.g., 3.0 mg. at p_H 5.2—5.9, 3.6—4.7 mg. at p_H 5.9—7.65. No correlation between numbers of bacteria and nitrogen fixation or soil reaction, or between soil reaction and disappearance of dextrose, was observed.

CHEMICAL ABSTRACTS.

Effect of green manures and crop residues on soil reaction. W. G. SACKETT, A. KEZER, I. W. FERGUSON, and J. C. WARD (Colo. Agric. Exp. Sta. Bull., 1928, No. 324, 1—31).—Green (barley) manure slightly increased the p_H of a silt loam containing 3.2% of calcium carbonate, but green manures, crop residues, and commercial fertilisers were not effective in changing the p_H of a soil rich in calcium carbonate from 7.7 to 6.0.

CHEMICAL ABSTRACTS.

Theory of growth factors [of soils]. E. A. MITSCHERLICH (Z. Pflanz. Düng., 1928, 7B, 345—352). O. LEMMERMAN and P. HASSE (Ibid., 352—361).—Adverse criticism of the theory (notably that of Gerlach and of Lemmermann) is discussed and the constancy of growth factors reaffirmed.

The technique of the Mitscherlich method is criticised. The dilution of the soil with sand introduces secondary factors with the system, and results are vitiated as the additional factors are not wholly incorporated in the calculation. Evidence is put forward to discredit the constancy of the growth factors.

A. G. POLLARD.

Wiessmann's method for determining the nutrient content of soils by pot experiments. K. MAIWALD (Z. Pflanz. Düng., 1928, 7B, 370—381).—The Wiessmann method (B., 1928, 279) is examined. The physiological effects of the mixing of soil and sand on plant growth are not fully considered in the original method. The growth curves in sand and on soil diluted with sand are not strictly comparable for the purpose of calculating the correction factor. The nutrient contents of the soil necessitate a different point of origin for the soil-sand growth curve. Further, the effect of the added fertiliser salts on the physical and chemical

conditions of different soil types may be such as to make comparison of the growth curves of little value.

A. G. POLLARD.

Cellulose in soil. WINOGRADSKY.—See V.

PATENTS.

Manufacture of mixed or complete fertilisers.

I. G. FARBENIND. A.-G. (B.P. 292,068, 2.6.28. Ger., 11.6.27).—Mixtures of two or more mineral acids, *e.g.*, nitric, sulphuric, and phosphoric acids, are atomised by ammonia or gases containing it. Portions of the acids may be replaced by their ammonium salts, and salts of fertilising value, *e.g.*, potassium salts, may be added in solution or in suspension. L. A. COLES.

Manufacture of high-grade fertilisers.

J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 295,477, 25.6.27).—Potassium chloride solution is treated with ground fluorspar, precipitated silica from a later stage of the process, and hydrochloric acid to yield a precipitate of potassium fluosilicate and a solution of calcium chloride. The precipitate is digested with ammonia, which regenerates the silica for further use and yields a solution of ammonium and potassium fluorides, which is then caused to react with the solution obtained by dissolving raw phosphate mineral in the minimum of nitric acid. Calcium fluoride is thus precipitated and returned to the first stage, and a solution of potassium and ammonium nitrates and phosphates is produced from which a high-grade fertiliser is obtained by evaporation. A. R. POWELL.

Manufacture of mixed fertilisers [containing ammonium nitrate]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 295,825, 5.8.27).—Non-caking mixed fertilisers are prepared by mixing solid salts of fertilising value with ammonium nitrate solution containing at least 92% of ammonium nitrate, maintained at a temperature high enough to keep it fluid for a sufficient time for the greater part of the water to evaporate during the mixing operation. L. A. COLES.

Solutions for treating grain.

I. G. FARBENIND. A.-G., Assees. of G. OSVALD (G.P. 447,601, 2.12.25. Swed., 20.8. and 3.10.25).—The solutions contain formaldehyde or its polymerides, together with alkali hydroxides, carbonates, bicarbonates, acetates, borates, phosphates, sulphites, hyposulphites, alkaline-earth hydroxides, ammonia, ammonium salts, aliphatic or aromatic amines, tanning agents, *etc.*, with or without the addition of mercury compounds, *e.g.*, mercuric cyanide, mercury salicylate, or mercuric compounds of phenols, carboxylic acids, or sulphonic acids. L. A. COLES.

Fungicides for the treatment of seeds.

I. G. FARBENIND. A.-G. (Austr. P. 107,003, 7.1.25).—Solutions are used containing alkali-soluble, complex, organic mercury compounds and cyanides or ferrocyanides, *e.g.*, 60 pts. of potassium ferrocyanide per 100 pts. of sodium mercuri-*p*-cresoxide. L. A. COLES.

Material for combating plant pests.

I. G. FARBENIND. A.-G. (G.P. 448,371, 10.3.25).—Activated, highly porous material, *e.g.*, charcoal or silica, impregnated with carbon disulphide is used. L. A. COLES.

[Hot-water pipe system for] drying of hay, straw, *etc.* C. LYON (B.P. 295,482, 4.7.27).

Nitrogen compounds (F.P. 622,266). **Dicalcium phosphate** (B.P. 293,942).—See VII.

XVII.—SUGARS; STARCHES; GUMS.

Formation of incrustation on evaporators [of beet-sugar factories]. V. STANĚK and P. PAVLAS (Z. Zuckerind. Czechoslov., 1928, 52, 545—560).—Incrustation formation in beet-sugar factories depends largely on the nature of the heating surface. Thus, on brass polished bright the percentage of incrustation and of suspension were 97.7 and 2.3, and on rough brass 94.6 and 5.46. Similarly, a copper-plated surface gave 67.3 and 32.7, and one which had been tinned 51.3 and 48.7. Calcium carbonate present as aragonite formed centres for the crystallisation of the calcium carbonate separated from a beet juice which had been over-carbonated; but calcium carbonate added in the amorphous form merely adhered to the heating surface, increasing the amount of the scaling. On the other hand, fine carbons (particularly "Norit") acted favourably in diminishing the amount of the scaling. A weak electric current between the heating surface and an electrode placed in the liquid exerted an unimportant, though detectable, effect. J. P. OGILVIE.

Determination of the degree of acidity (p_H value) of sugars. P. HONIG and P. C. NICOLA (Arch. Suikerind. Nederl.-Indië, 1928, 36, 6—13).—In determining the p_H value of sugars, 20 g. were dissolved in 30 c.c. of neutral water, and 10 c.c. of this solution with 0.5 c.c. of indicator compared with the standard tubes in the usual way. Values from 5.4 to 6.6 were found for various Java plantation white (1927) sugars, those made in sulphitation factories being, in general, less acid than those made by carbonation. A Dutch semi-refined beet sugar gave p_H 6.65; French beet sugar (1925 campaign) 5.20, and a sample of refined sugar 6.40. It was found that the p_H generally falls as the concentration of the sugar solutions examined is increased. J. P. OGILVIE.

Determination of alcoholic extractive in gum benzoin. T. N. BENNETT and C. F. BICKFORD (J. Assoc. Off. Agric. Chem., 1928, 11, 386—388).—The method given in U.S.P.X. is unsatisfactory as the drying of the alcohol extract at 110° involves the loss of certain constituents, notably benzoic acid. Five alternative methods were tried, of which the simplest and quickest, which is also accurate, consists in extracting 2 g. of the sample, placed in a tared thimble, with 95% alcohol containing about 0.5 g. of sodium hydroxide for 5 hrs. in a continuous extraction apparatus. The thimble is dried, weighed, and the alcoholic extractive plus water is calculated by difference, the latter being determined by the xylol distillation method. F. R. ENNOS.

Basic lead acetate. SOMMER.—See VII.

XVIII.—FERMENTATION INDUSTRIES.

Protease and amylase of *Aspergillus oryzae* K. OSHIMA (J. Coll. Agric. Hokkaido Imp. Univ., 1928, 19, 135—243).—Amylase and protease are obtained on synthetic and natural culture media simultaneously and in optimal quantity after two

days' growth; on artificial culture media a mixture of casein or peptone and starch or dextrose is a stimulant. Amylase and protease are extra-cellular after sporulation; different species of *Aspergillus* show variation in activity of the enzyme. The protease is more stable at neutrality and 40° than trypsin, is most resistant to heat at p_H 6.4, has optimal activity at 50° and p_H 5.2–5.3, and digests natural proteins and peptones. The amylase is most active at p_H 4.8–5.2, and most stable towards heat at p_H 6.4; it retains its activity below 40° at neutrality. Its action is inhibited by sodium chloride and by alcohol. Unlike that of the protease, the reaction velocity follows the unimolecular formula. CHEMICAL ABSTRACTS.

Amylase content of raw grain and its utilisation in grain distilleries. STAIGER (Chem.-Ztg., 1928, 69, 679–680).—By modifying the process of mashing, the amount of amylase in rye and wheat is sufficient to saccharify completely the starch present in the corns. No malt requires to be added and the corn does not require to be germinated. The diastatic power of barley is much lower and complete saccharification is rarely obtained without added malt. If the worts, obtained by saccharifying the raw grain with its own amylase, are fermented, the resulting brandy is of a mild nature and the content of fusel oil is low. The process is economical, saving both time and fuel, owing to the omission of the customary cooking of the raw grain. C. RANKEN.

PATENTS.

Yeast stimulants and process of using them. T. B. WAGNER (U.S.P. 1,680,827, 14.8.28. Appl., 2.6.23. Renewed 13.1.28).—A mixture of amylaceous material and the dried, soluble constituents of corn steepwater is added to dough. F. G. CLARKE.

Producing a diastatic product. K. WOYENAKA and T. OKOCHI, ASSTS. to TAKAMINE FERMENT CO. (U.S.P. 1,680,926, 14.8.28. Appl., 9.1.23).—A suitable culture medium is impregnated with a solution of sodium salicylate and fluoride, sterilised by steaming, and treated with dilute mineral acid. The mass is inoculated with spores of *Aspergillus oryzae* and incubated. B. FULLMAN.

Preparation of yeast. G. O. W. HEIKENSKJÖLD, ASSR. to AKTIEBOLAGET BÄSTA (U.S.P. 1,680,043, 7.8.28. Appl., 6.2.26. Sved., 28.3.25).—See F.P. 614,037; B., 1927, 921.

Collecting inorganic potassium salt, betaine salt, and glutamic acid from waste liquor produced in distilling alcohol from fermented beet molasses. Y. TAKAYAMA (U.S.P. 1,681,379, 21.8.28. Appl., 12.1.27).—See B.P. 288,390; B., 1928, 424.

Mineral oils from peat etc. (B.P. 270,349).—See II. **Fats from cacao beans** (U.S.P. 1,680,943).—See XII. **Curds into cheese** (U.S.P. 1,676,121).—See XIX.

XIX.—FOODS.

Fat determination in milk and cream confectionery. H. BARSCH (Chem.-Ztg., 1928, 52, 659).—An improved procedure, based on the methods of Gottlieb and Röse (cf. von Gahrtz, Pharm. Zentr., 1927, 68, 177–179) and of Kuhlmann and Grossfeld (B., 1926,

252) for the determination of fat in confectionery is proposed, adopting trichloroethylene as solvent for the fat. E. LEWKOWITSCH.

Determination of milk solids in mixed feeds. A. B. DAVIS (J. Assoc. Off. Agric. Chem., 1928, 11, 410–417).—The various methods available are summarised. The qualitative tests depend on the identification of lactose or casein, or on the microscopic appearance of the feed. In the quantitative methods lactose is determined by Fehling's solution after fermentation with yeast or treatment with basic lead acetate, or from the total reducing power combined with the yield of insoluble osazones. The percentage of milk solids is calculated by means of a lactose-milk solids factor; reliable results are obtainable, especially if a sample of buttermilk solids is available for determination of the appropriate factor. F. R. ENNOS.

Preparation of butter samples for analysis. J. A. NEWLANDER and H. B. ELLENBERGER (Vermont Sta. Bull., 1927, No. 263, 31 pp.).—Two standard methods are suggested. In 90% of cases duplicate determinations should agree to within 0.05%.

CHEMICAL ABSTRACTS.

Moisture content of oleomargarine. R. H. KERR (J. Assoc. Off. Agric. Chem., 1928, 11, 382–386).—Oleomargarine containing animal fats falls into two classes—that intended for use as a butter substitute which contains 8–14% of moisture, and that used for cooking or baking only with rather less moisture. When made from vegetable fats and skimmed milk, it has a moisture content similar to the former class.

F. R. ENNOS.

Effect of various rations on storage quality of eggs. R. M. SHERWOOD (Texas Agric. Exp. Sta. Bull., 1928, No. 376, 12 pp.).—Hens receiving a mash containing 9% (but not 20–30%) of 43% protein cottonseed meal yielded eggs which remained of a good colour in cold storage. Fresh green feed improves the storage quality of eggs. CHEMICAL ABSTRACTS.

Studies of tomato quality. II. Effect of soil moisture on percentage of dry matter in the fruit. R. E. BROOKS and J. H. MACGILLIVRAY (J. Assoc. Off. Agric. Chem., 1928, 11, 389–393).—The percentage of dry matter of a tomato fruit varies inversely with that of the soil moisture; an increase in the latter results in a greater number, weight, and volume of the fruit formed.

F. R. ENNOS.

Determination of water in dried prunes. E. H. WIEGAND and D. E. BULLIS (Oregon Agric. Coll. Exp. Sta. Circ., 1927, 82, 1–8).—The hashed prune flesh (20 g.) and toluene (200 c.c.) are boiled vigorously for 30 min. in a distillation flask connected to a graduated tube serving as the receiver of a reflux condenser, the amount of water being read directly from the graduations. Samples contained 15–25 (± 0.25)% of water; those containing below 20% of water remained in good condition under storage tests. CHEMICAL ABSTRACTS.

Detection of artificial colouring matters in fruit juices and the like. A. DE KROES and A. RECLAIRE (Chem. Weekblad, 1928, 25, 525–528).—The usual tests have been carried out with pure fruit juices, and it is found that positive results may be obtained in most

cases; great care is therefore necessary in deciding whether artificial colouring matters have been added. The wool test of Arata gives more satisfactory results if a second treatment of the solution with the fibre is given, the solution being acidified for the second.

S. I. LEVY.

Application to meat and meat food products of a rapid-boiling short-digestion method for determination of protein. H. R. McMILLIN (J. Assoc. Off. Agric. Chem., 1928, 11, 408—410).—Determinations of protein ($N \times 6.25$) in meat and meat food products by this method (cf. Shedd, B., 1927, 954) are in good agreement with those carried out by the official Gunning method, and result in the saving of 1—3½ hrs. in the analysis as compared with the latter. F. R. ENNOS.

Nutritive value of alba blood as a source of protein. S. M. HAUGE (J. Assoc. Off. Agric. Chem., 1928, 11, 398—403).—Alba blood—a by-product recovered from spent printers' rollers by removing the glycerol, dissolving the residual gelatin etc. in water, and drying—is inadequate both as a sole source of protein or as a supplement to corn. It also has a repressing effect on growth and should not be used to increase the protein content of tankages and meat by-products used as feeding stuffs. F. R. ENNOS.

Vitamin content of canned spinach. E. PIERSON (S. Dakota Agric. Exp. Sta. Ann. Rep., 1926, 23).

Composition and nutritive value of Philippine food fishes. A. VALENZUELA (Philippine J. Sci., 1928, 36, 235—242).

Composition of Philippine pineapples. A. H. WELLS, F. AGCAOILI, II. TAQUIBAO, and A. VALENZUELA (Philippine J. Sci., 1928, 36, 157—184).

PATENTS.

Transforming pieces of curds into cheese by treatment with salts. R. VON DER HEIDE (U.S.P. 1,676,121, 3.7.28. Appl., 25.5.26. Ger., 12.5.25).—The pieces of curds are treated with bacteria, salts, and compounds of the ammonium, amide, or imide groups, e.g., ammonium carbonate, lactate, and chloride at 15—30°, or a mixture of 50% of amino-acids, 40% of common salt, 4% of magnesium chloride, 4% of calcium phosphate, 1% of iron salts, and 1% of manganese salts. F. R. ENNOS.

Preparation of a beverage. C. HORIX (B.P. 294,003, 25.8.27).—Maté leaves are steeped in water at 100° for 5—15 min. and the infusion is filtered, cooled, freed from air in a vacuum chamber, and impregnated with carbon dioxide. It is finally sealed in closed bottles, and flavouring added if desired. F. R. ENNOS.

Production of antirachitic substances. A. J. PACINI, Assr. to C. M. RICHTER (U.S.P. 1,681,120, 14.8.28. Appl., 24.8.27).—Growth-producing substances are partially converted into antirachitic products by subjecting them to rays of longer wave-length than 3022 Å. F. G. CLARKE.

Refrigerating and preserving comestibles. P. W. PETERSEN (U.S.P. 1,681,009, 14.8.28. Appl., 23.6.23).—The comestibles are washed in a solution of a germicidal agent, e.g., sodium hypochlorite, then rapidly frozen while still wet, and given a coating of ice.

H. ROYAL-DAWSON.

Antirachitic product and process. II. STEENBOCK, Assr. to WISCONSIN ALUMNI RES. FOUNDATION (U.S.P. 1,680,818, 14.8.28. Appl., 30.6.24).—See B.P. 236,197; B., 1925, 735.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

d-Borneol and cis-terpin from distillation residues of sulphate-turpentine oil. W. QVIST (Biochem. Z., 1928, 197, 476—481).—From the fraction of the residues distilling at 120—145°/15 mm. a yield of 0.35% of cis-terpin was obtained. A yield of 2.1% of a mixture of i-borneol with 11—15% of d-borneol was obtained by distilling the residues in superheated steam, fractionating the dried distillate at ordinary pressure, and crystallising the solid which separated from the fraction which boiled at 200—230°. The borneol was found to be free from isoborneol. Since borneol derived from sulphite-cellulose manufacture is levorotatory the different activities may be due to different methods of preparation, but more probably to the different woods used as raw materials. W. MCCARTNEY.

Yeast method for silver proteins. II. WALES (J. Assoc. Off. Agric. Chem., 1928, 11, 396—398; cf. Taylor, B., 1927, 858).—The action of silver nitrate on yeast differs entirely from that of silver protein, and hence the inhibitory action of the latter on the growth of yeast does not appear to be due to the concentration of the silver ion. F. R. ENNOS.

Assay of trional tablets. L. E. WARREN (J. Assoc. Off. Agric. Chem., 1928, 11, 404—407).—Powdered trional tablets are extracted with chloroform either by maceration and decantation of the solvent through a filter, or in a Bailey or Soxhlet extractor. The solvent is evaporated at ordinary temperature by a current of air, and the residue is dried to constant weight in a desiccator over sulphuric acid. The results obtained are on the average 1½% too high. F. R. ENNOS.

Determination of essential oils in drugs. H. KAISER and K. EGGENSERGER (Pharm. Ztg., 1928, 73, 1036; cf. Bauer, B., 1928, 691).—The salt which separates during the distillation of the pentane contains some oil, which must be determined by the difference in weight after further extraction with pentane.

S. I. LEVY.

Preparation of 2-phenylquinoline-4-carboxylic acid [atophan]. F. CHEMNITZ (Pharm. Zentr., 1928, 69, 549—551).—Practical details for the preparation of pyruvic acid and benzylidene-aniline, and their condensation to form atophan, are given. C. HOLLINS.

Essential oils of the *Thuja* species from the Crimea. B. RUTOVSKI and S. BUSSE (Trans. sci. chem.-pharm. Inst., Moscow, 1924, [10], 6—18; Chem. Zentr., 1927, II, 1760—1761).—The oils were obtained from the twigs and leaves. *Thuja gigantea*, Nutt., gave 1.12% of oil having d_{20}^{20} 0.9125, α_D -1.50°, n_D^{20} 1.4550, acid value 0.38, ester value 12.79, ester value after acetylation 33.02, carbonyl number (Walter) 261.3, solubility in 80% alcohol 1 in 1 vol. The oil contains sabinene, α -thujone, thujyl alcohol, and an aldehyde which on oxidation gave an alcohol (diphenylurethane, m.p. 52°). α -Pinene and fenchone could not be detected. *Thuja occidentalis*, L., gave 0.082% of oil having d_{20}^{20} 0.89934, α_D +15.59°, n_D 1.4810, acid value 0.54, ester value 23.26, ester value

after acetylation 49.55, carbonyl number 43.7, solubility in 90% alcohol 1 in 4.5 vols., and containing d - α -pinene and thujone. *Thuja orientalis*, L., gave 0.12% of oil having d_{20}^{25} 0.87955, $\alpha_D + 13.78^\circ$, n_D^{20} 1.4792, acid value 0.34, ester value 15.14, ester value after acetylation 37.2, carbonyl number nil, solubility in 90% alcohol 1 in 8.5 vols. The oil contains d - α -pinene, a sesquiterpene alcohol, and probably caryophyllene. E. H. SHARPLES.

Hyssop oil from the Crimea. B. RUTOVSKI and I. VINOGRADOVA (Trans. sci. chem.-pharm. Inst., Moscow, 1924, [10], 22—23; Chem. Zentr., 1927, II, 1761).—*Hyssopus officinalis*, L., gave an oil having d_4^{20} 0.9573, $\alpha_D - 19.29^\circ$, n_D 1.4805, acid value 4.03, ester value 35.86, ester value after acetylation 98.02, solubility in 90% alcohol 1 in 0.4 vol. The following compounds were identified in the oil: α - and β -pinene, *l*-pinocampphone (*tribromopinocampphone*, m.p. 93—95°), *l*-pinocampheol (m.p. 56—57°, d_4^{20} 0.9509, $\alpha_D - 45.63^\circ$, n_D^{20} 1.4888), an aldehyde (d_4^{20} 0.9446, $\alpha_D - 13.0^\circ$, semicarbazone, m.p. 201.5—203°), and acetic acid. Cineole could not be detected. E. H. SHARPLES.

Fennel oil from the Crimea. B. RUTOVSKI and P. LEONOV (Trans. sci. chem.-pharm. Inst., Moscow, 1924, [10], 64—67; Chem. Zentr., 1927, II, 1761).—The oil, obtained in a yield of 0.61%, had d_4^{20} 0.9705, $\alpha_D + 12.15^\circ$, n_D 1.5405, acid value nil, solubility in 80% alcohol 1 in 6.3 vols., and contained anethole, phellandrene, and methylchavicol. Fenchone could not be detected. E. H. SHARPLES.

Fennel oil. B. RUTOVSKI and L. ZÜRICH (Trans. sci. chem.-pharm. Inst., Moscow, 1924, [10], 69—70; Chem. Zentr., 1927, II, 1762).—Extraction with ether gave 3.03% of essential oil having d_4^{20} 0.9430, $\alpha_D + 9.35^\circ$, n_D^{20} 1.5384, and acid value 0.94. Oil obtained by direct distillation had d_4^{20} 0.9653, $\alpha_D + 17.05^\circ$, n_D^{20} 1.5312, and acid value nil. E. H. SHARPLES.

Constants of some Crimean and Caucasian essential oils. B. RUTOVSKI and I. VINOGRADOVA (Trans. sci. chem.-pharm. Inst., Moscow, 1924, [10], 88—89; Chem. Zentr., 1927, II, 1762).—The constants of oils distilled in 1923 are tabulated. E. H. SHARPLES.

Essential oils from the berries of *Juniperus excelsa*, MB. B. RUTOVSKI and I. VINOGRADOVA (Trans. sci. chem.-pharm. Inst. Moscow, 1924, [10], 51—61; Chem. Zentr., 1927, II, 1761).—1.1% of oil was obtained from the berries of *J. excelsa*, MB., from the Crimea. It contained α -pinene, borneol, isoborneol, and acetic acid. β -Pinene could not be detected. E. H. SHARPLES.

Essential oil of "Hiba" wood and its relation to the resistability of the wood against corrosion. S. UCHIDA (J. Soc. Chem. Ind. Japan, 1928, 31, 501—503).—By steam distillation of sawdust (water content 27.73%) of "Hiba" wood (*Thujopsis dolabratus*, Sieb et Zucc), 1.08% of an oil was obtained having d_{15}^{25} 0.9574, n_D^{20} 1.5110, $[\alpha]_D - 26.72^\circ$ in chloroform, acid value 5.62, ester value 11.97, and saponif. value 17.59. The oil chiefly contains a tereyclic inactive sesquiterpene, $C_{15}H_{24}$, having b.p. 261—262°, d_4^{25} 0.9458, n_D^{20} 1.5055, and M_D 64.09°. The oil is toxic to wood-destroying fungi, e.g., *Merulius lachrymans*, Fr., and *Polyporus gilvus*, Schw.; hence the property of the wood to resist corrosion. K. KASHIMA.

Essential oil of leaves of "Hiba." S. UCHIDA (J. Soc. Chem. Ind. Japan, 1928, 31, 491—501).—Steam distillation of leaves of "Hiba" yielded 0.83% of oil having d_4^{25} 0.8857, n_D^{20} 1.4729, $[\alpha]_D + 27.67^\circ$, acid value 3.76, saponif. value 32.68, ester value 29.12, and ester value after acetylation 113.2. Compounds isolated from the oil are: undecic acid, sabinene, dipentene, borneol, sabinol, bornyl and sabinyl acetates, a dicyclic sesquiterpene, $C_{15}H_{24}$, having b.p. 270—280°, d_4^{25} 0.9232, n_D^{20} 1.4949 (additive product with 2HCl), a monocyclic sesquiterpene alcohol, b.p. 290—300°, d_4^{25} 0.9328, n_D^{20} 1.5049, M_D 70.60, and a tetracyclic diterpene, $C_{20}H_{32}$, having b.p. 336°, d_4^{20} 0.19629, n_D^{20} 1.5131, M_D 84.92. The oil consists of about 50% of terpenes (sabinene and a small amount of dipentene), 22% of terpene alcohols (sabinol and borneol, the former predominating), 10% of esters (mainly sabinyl acetate, together with bornyl acetate), 13% of diterpene, 3% of sesquiterpene alcohol, 2% of sesquiterpene, and 0.1% of undecic acid. K. KASHIMA.

Essential oil of leaves of "Hinoki." S. UCHIDA (J. Soc. Chem. Ind. Japan, 1928, 31, 650—659).—By steam distillation of leaves of "Hinoki" (*Chamaecyparis obtusa*, Endl) 1.16% of an essential oil was obtained having d_{15}^{25} 0.9053, $\alpha_D^{25} + 41.25^\circ$, n_D^{20} 1.4794, acid value 0.60, ester value 75.37, and ester value after acetylation 100.2. The following compounds were isolated: a crystalline sesquiterpene acid ("Hinoki" acid), $C_{14}H_{23} \cdot CO_2H$, m.p. 166° (corr.) (silver salt, amorphous; additive product, m.p. 119°, with 1HCl); also d - α -pinene; d -limonene; d -borneol; bornyl acetate and nonoate: a tricyclic *l*-sesquiterpene ($C_{15}H_{24}$), having b.p. 265—268°, d_4^{20} 1.5009, $[\alpha]_D - 15.67^\circ$ in alcohol, M_D 64.21° (additive product with 1HCl); cadinene: a dicyclic *d*-sesquiterpene alcohol ($C_{15}H_{26} \cdot OH$), having b.p. 290—295°, d_4^{20} 0.9607, n_D^{20} 1.5020, $[\alpha]_D + 18.0^\circ$ in chloroform, M_D 68.22° (benzoate); and a tetracyclic inactive diterpene ($C_{20}H_{32}$), b.p. 340—343°, d_4^{20} 0.9632, n_D^{20} 1.5190, M_D 85.21. About 40% of the oil consists of a mixture of d -limonene and d - α -pinene, the former predominating, about 25% esters (bornyl acetate and trace of nonoate), 20% of sesquiterpenes (mainly tricyclic and trace of cadinene), about 10% of alcohols (mainly the dicyclic *d*-sesquiterpene alcohol), and about 5% of tetracyclic inactive diterpene. K. KASHIMA.

Essential oil of leaves of "Sawara." S. UCHIDA (J. Soc. Chem. Ind. Japan, 1928, 31, 642—650).—By steam distillation of leaves of "Sawara" (*Chamaecyparis pisifera*, Endl) 1.07% of an essential oil was obtained having d_4^{20} 0.8939, n_D^{20} 1.4710, $\alpha_D^{20} + 12.55^\circ$, acid value 1.50, saponif. value 55.20, ester value 53.70, ester value after acetylation 64.49. The following compounds were isolated: pelargonic acid, undecic acid, d - α -pinene, dipentene (additive product, $C_{20}H_{32} \cdot HCl$), borneol, bornyl acetate and formate, a sesquiterpene alcohol, $C_{15}H_{25} \cdot OH$, b.p. 290—300°, n_D^{20} 1.5002, and a tetracyclic diterpene, $C_{20}H_{32}$, b.p. 320—335°, d_4^{20} 0.9452, n_D^{20} 1.5125, M_D 86.54. About 75% of the oil consists of terpenes (α -pinene, traces of dipentene), 20% of esters (bornyl acetate, traces of the formate), 3% of alcohol, 2% diterpene, and traces of the acid. K. KASHIMA.

Extract of gum benzoin. BENNETT and BICKFORD.—See XVII.

PATENTS.

Obtaining germ gland hormones from vegetable organisms. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (B.P. 271,492, 19.5.27. Ger., 20.5.26).—The lipid-soluble parts are extracted from vegetable organisms (rhubarb, potato, etc.) by organic solvents, the extract after being concentrated is treated with water or a water-miscible solvent, and the residue is extracted with ether. B. FULLMAN.

Parathyroid product. L. BERMAN (U.S.P. 1,680,660, 14.8.28. Appl., 11.8.25).—Comminuted parathyroid gland is extracted with aqueous metaphosphoric acid. The extract is neutralised with sodium hydroxide and filtered, the filtrate from which is rendered alkaline to form a phosphate gel, filtered, and the filtrate neutralised. B. FULLMAN.

Preparation of bile acid salts of cinchona alkaloids. CHEM. WORKS FORMERLY SANDOZ (B.P. 282,356, 8.11.27. Switz., 17.12.26).—Equimolecular salts of quinine, quinidine, dihydrocupreine ethylether with cholic and deoxycholic acids are described. C. HOLLINS.

Tobacco substitute with therapeutical properties. F. K. CHISHOLM (U.S.P. 1,680,860, 14.8.28. Appl., 2.12.26. Austral., 18.1.26).—Natural eucalyptus leaves are semi-dried, rolled, macerated in a solution containing 10% of a hygroscopic and 10% of a flagrating substance, kept at 37·8–93·3° for 3 hrs., subjected to a pressure of 2 tons/in.² at not above 100°, and the pressed mass is cured by ageing. B. FULLMAN.

Preparation of emetine. CHEM. FABR. VORM. SANDOZ (B.P. 291,088, 2.5.28. Switz., 27.5.27).—See U.S.P. 1,670,059; B., 1928, 502.

Production of bactericidal and therapeutic agents. [Biological stains.] R. W. FRENCH and W. C. HOLMES (B.P. 295,605, 8.2.27).—See U.S.P. 1,661,568; B., 1928, 328.

Manufacture of 3-bromo-2-hydroxypyridine-5-arsinic acid. A. BINZ and C. RÄTH (U.S.P. 1,680,587, 14.8.28. Appl., 17.12.26. Ger., 21.12.25).—See B.P. 263,142; B., 1927, 734.

Esters of unsaturated acids. W. MINNICH, ASSR. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,680,799, 14.8.28. Appl., 3.2.28. Switz., 19.11.23).—See B.P. 243,510; B., 1926, 141.

Manufacture of ointments. F. MERZ (MERZ & Co.) (B.P. 268,719, 23.9.26. Ger., 30.3.26).—See G.P. 443,756; B., 1928, 548.

Calcined phosphates (G.P. 447,665).—See VII. **Antirachitic substances** (U.S.P. 1,681,120).—See XIX.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Sensitisation of chrome gelatin [photographic plates] to orange and red. G. O. T. HOOFT (Z. wiss. Phot., 1928, 25, 394–400).—Chrome gelatin plates which are sensitive to orange and red light may be prepared by bathing the gelatin film before treatment with the potassium dichromate solution in any of the following dye solutions:—Pinatype Green D.A. 88778 (the later preparations of this dye do not act), Kiton Green, Diamond Green, Brilliant Acid Green, Fuchsin Red, Brilliant Silk Blue, and Methylene Blue.

J. W. GLASSETT.

Chloramine for destruction of last traces of sodium thiosulphate in photographic plates or papers. J. M. EDER (Z. wiss. Phot., 1928, 25, 401–402).—A 0·2% solution of chloramine, which hydrolyses in aqueous solution to give *p*-toluenesulphonamide, sodium chloride, and active oxygen, is suggested for the rapid oxidation of the last traces of sodium thiosulphate. The developed silver image is attacked by stronger solutions or prolonged action. J. W. GLASSETT.

PATENTS.

Manufacture of diazo-types. F. VAN DER GRINTEN (B.P. 294,972, 3.5.27).—In order to prevent discoloration of the whites in diazo-types a reducing agent is incorporated with the sensitising layer or is added during or after development, preferably together with oxidation anticatalysts such as *m*-phenylenediamine or pyrogallol. When the coupling component is present with the diazo compound (for development with alkali alone) it is protected from premature coupling by combination with a suitable agent; *e.g.*, phloroglucinol is combined with hydroxylamine to give the trioxime of triketocyclohexane, and bisulphite compounds, cyanohydrins, hydrazones, etc. of the ketonic forms of phenolic components may also be used. Various other alicyclic derivatives and even those diazo compounds which generate phenols in alkaline solution serve the same purpose. Ammonia gas is suitable for developing the image, but an alkaline bath, or a not too dry alkaline powder, such as sodium stannate, may be used. Development by heat alone is feasible when the inhibiting acid is one which is readily decomposed by heat. Bleeding is avoided by addition of salts such as barium chloride. In the examples the sensitised layer contains the following ingredients: diazotised *p*-aminodimethylaniline, oxalic acid, barium chloride (developed with alkaline Naphthol AS containing 0·4% of formaldehyde, for black); *p*-dimethylaminobenzenediazonium chlorostannate, glucose, mannitol, ammonium citrate, hydroxylamine sulphate, and a trace of pyrogallol (developed with phloroglucinol, resorcinol, ammonia, sodium acetate, and sodium citrate, for black); 3-chloro-4-dimethylaminobenzenediazonium chlorostannate, sodium sulphate, hexamethylenetetramine, glyceraldehyde (developed with phloroglucinol for dark brown); 5-diisoamylamino-*m*-xylene-2-diazonium chlorostannate, phloroglucinol, hydroxylamine hydrochloride (developed with sodium hydroxide and formaldehyde, for black); triketocyclohexane trioxime, *p*-dimethylaminobenzenediazonium chlorostannate, oxalic acid, ammonium lactate, hexamethylenetetramine, glyceraldehyde, and traces of pyrogallol and *m*-phenylenediamine (developed with ammonia gas for black, or if sodium acetate be substituted for the oxalic acid development by a hot smoothing iron gives a black). C. HOLLINS.

Photographic film. F. F. RENWICK, ASSR. to DU POST-PATHE FILM MANUF. CORP. (U.S.P. 1,680,635—6, 14.8.28. Appl., 20.8.25).—(A) The backing of a cellulose nitrate film contains a protein, *e.g.*, gelatin, and possibly also a cellulose ester. (B) The backing of the film, which is intended for cinematography, is electropositive with respect to the body portion, and comprises a water-insoluble protein, *e.g.*, casein, free from pigmentary and other granular materials.

F. G. CLARKE.

Colour photography. W. V. D. KELLEY and D. TRONOLONE, Assrs. to KELLEY COLOR LABORATORY, INC. (U.S.P. 1,674,174, 19.6.28. Appl., 11.1.24).—A coloured photograph is obtained by first forming an image in one section of the coating, toning the image to a given colour, and, without fixing, forming a second image in the same coating, but in a different section thereof, rendering the colloid surrounding the reduced light-sensitive salt of the second image capable of adsorbing an azo or acid dye, dyeing to a colour complementary to that of the first image, and finally fixing both images in the usual way. A. R. POWELL.

Colour photography. W. V. D. KELLEY (U.S.P. 1,674,175, 19.6.28. Appl., 9.11.25).—A coloured photograph composed of three silver images coloured and developed separately is claimed. A. R. POWELL.

Photographic reproduction of objects in their natural colours or in monochrome. S. M. PHELPS (U.S.P. 1,673,300, 12.6.28. Appl., 18.12.23).—A matrix-film is prepared containing a dye combined with a mordant which is sensitive to light. It is then exposed behind a negative and the liberated dye transferred to another surface. F. G. CLARKE.

XXII.—EXPLOSIVES; MATCHES.

Laws of combustion of colloidal powders. II. MURAOUR (Compt. rend., 1928, 187, 374—375; cf. A., 1927, 736).—The total area of the pressure-time curve, which is characteristic for a given explosive powder, is not modified if the powder is partly substituted by one of different intensity, whatever the temperature of the gas emitted by the latter. The gaseous envelope surrounding the particles of the powder acts solely by reason of its pressure and not by its temperature. J. GRANT.

PATENTS.

Production of trinitrotoluene. A.-G. LIGNOSE (B.P. 294,625, 13.2.28. Ger., 29.7.27).—Molten dinitrotoluene at 80—95° is allowed to flow into mixed acid at an initial temperature of 80°. The rate of inflow should be regulated to prevent the temperature of the reaction mixture from exceeding 95°. Nitration is complete in 3—4 hrs. S. BINNING.

Uninterruptedly washing nitroglycerin, nitroglycol, and like esters. A. SCHMID and J. MEISSNER (B.P. 284,702, 28.1.28. Ger., 5.2.27).—Continuous washing of nitroglycerin or nitroglycol is effected by forming an emulsion of the nitric ester and the washing solution by compressed air in such a way that intermingling of the whole contents of the washing vessel is prevented. A suitable apparatus is a vessel divided by screens into superposed compartments up through which the emulsion passes step by step. S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Influence of sulphite waste liquor effluent on the degree of acidity of river water. E. ÖMAN (Papier-Fabr., 1928, 26, 529—532).—The effect of sulphite liquor effluent on river water depends on the natural degree of acidity of the latter and on its capacity for acting as a buffer liquid in virtue of the effect of dissolved carbonates. A dilution of waste liquor in the

ratio 1:350 changed the p_H of river water from 6.6 to 4.8, whilst a dilution of 1:3000 resulted only in the change 6.6 to 6.4; such changes are of no importance. Comparison of the p_H of a river water-waste-liquor mixture with those of fruit juices, wine, beer, etc. indicates that water with a degree of acidity corresponding to p_H 4 or over is harmful neither to flora nor fauna. Prolonged storage of a dilute solution of waste liquor results in a gradual but small increase of acidity, which is attributed to the oxidation of sulphurous to sulphuric acid. B. P. RIDGE.

Corrosion of condenser tubes. MAY.—See X. Cresol soaps. KOGAN.—See XII.

PATENTS.

Prevention of boiler incrustation. W. LAZARUS (B.P. 294,224, 29.10.27. Ger., 21.7.27. Addn. to B.P. 283,517; B., 1928, 732).—After the addition of soot etc. to the feed water, alkaline-earth compounds, e.g., barium hydroxide or carbonate, are added instead of alkali, the amount necessary for a period being calculated and added all at one time. W. G. CAREY.

Emulsions for use in preventing corrosion or scale-formation in boilers. SOC. DES PERFECTIONNEMENTS APPL. À L'IND. (B.P. 285,057, 3.2.28. Fr., 10.2.27).—Vegetable seed cake from which most of the oil has been extracted (e.g., linseed cake) is treated with hot water to obtain the mucilaginous material. W. G. CAREY.

Purification of water. R. B. DERR, Assr. to NAT. ALUMINATE CORP. (U.S.P. 1,680,506, 14.8.28. Appl., 20.9.27).—Water containing less than 50 p.p.m. of magnesium sulphate is purified by mixing with it a water-softening compound and sodium aluminate, and accelerating the formation of a coagulant from the aluminate by adding a substance capable of producing magnesium ions and sulphate ions. W. G. CAREY.

Treatment of water for household and industrial purposes. J. W. M. BOURGOGNION and C. L. PHILIPS (B.P. 271,079, 10.5.27. Holl., 11.5.26).—Soft water is treated with carbon dioxide and then with calcium or magnesium carbonate so that it acquires the property of depositing a layer of calcium or magnesium carbonate upon tubes etc. through which it passes, the sum of the fixed and free carbon dioxide at the completion of the treatment preferably being 58.9 mg./litre. L. A. COLES.

Treatment of water. A. R. MÖBERG, Assr. to NAT. ALUMINATE CORP. (U.S.P. 1,679,777, 7.8.28. Appl., 23.12.26).—To the water is added aluminium sulphate and relatively small quantities of sodium aluminate. H. ROYAL-DAWSON.

Water purifier. J. B. GAIL and N. ADAM (U.S.P. 1,680,867, 14.8.28. Appl., 11.4.21. Fr., 15.4.20).—See B.P. 180,420; B., 1922, 565 A.

Sewage purifying device. S. BURGESS. From H. E. ELROD (B.P. 295,863, 24.10.27).

Water purifying apparatus. J. MUCHKA (B.P. 295,529, 31.10.27. Austr., 28.9.27).

Electric apparatus for preventing and removing deposits in water-evaporating vessels. L. ERÉNYI (B.P. 265,580, 2.2.27).

Moth-proofing (B.P. 274,425).—See V.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

OCT. 26, 1928.

I.—GENERAL; PLANT; MACHINERY.

Effect of pressure on fundamental filtration equation when solids are non-rigid or deformable.

D. R. SPERRY (Ind. Eng. Chem., 1928, 20, 892—895).—For filtration under constant pressure the rate of flow per unit area is given by $\sqrt{\omega PT}$, approx., where ω is a constant for a given suspension, P is the pressure, and T the time. The value ωP can be calculated from the time-discharge curve if two points on it and the pressure are known. Time-discharge curves were taken for given mixtures under several different pressures and the value of ωP for each was computed. For rigid solids the relation of ωP to P should be (on logarithmic paper) a straight line at an angle of 45° . If the solids are not rigid the angle is less and the exponent of P is given by the tangent of the angle. This value as found varied from 0.87 for starch down to 0.115 for fuller's earth. The effect of increase of pressure on the rate of filtration of the latter is therefore very slight. C. IRWIN.

Quantitative relations of the counter-current washing process.

L. SILBERSTEIN (Ind. Eng. Chem., 1928, 20, 899—901).—In the systematic washing of an insoluble solid, if p is the concentration of the solute in the adherent mother liquor, a the weight of this, and b the weight of liquor transferred at each washing, then, writing $\alpha = a/(a+b)$, $\beta = b/(a+b)$, and $s = \alpha\beta \leq \frac{1}{2}$, it is shown that if 3 tanks are used the concentration of the liquor in the third tank when equilibrium has been established is $\alpha^3 p / (1 - 2s)$. If 4 tanks are used it is $\alpha^4 p / (1 - 3s) + s^2$, and with 5 tanks $\alpha^5 p / [(1-s)(1-3s)]$. The data also permit of the calculation of the concentration of any other of the liquors used. C. IRWIN.

Conditions governing extraction of a solution by an immiscible solvent.

E. L. SMITH (J.S.C.I., 1928, 47, 159—160 r).—A discussion of the general principles involved in the extraction of a solute from its solution by an immiscible solvent. It is shown that if the number of extractions and total volume of extracting solvent are fixed, the maximum extraction is obtained by dividing the extracting solvent into equal parts.

Recommended specifications for analytical reagent chemicals.

W. D. COLLINS, H. V. FARR, J. ROSIN, G. C. SPENCER, and E. WICHES (Ind. Eng. Chem., 1928, 20, 979—983).

Electrification in dust clouds. BLACKTIN.—See II.

PATENTS.

Furnace. F. MENNE, Assr. to H. F. HOEVEL (U.S.P. 1,680,468, 14.8.28. Appl., 20.5.27. Ger., 9.10.25).—

A furnace with a longitudinal chamber is provided with a loose hearth from one end of which project spaced cooling pipes, forming a grid through which loose material can drop. B. M. VENABLES.

Retorts or furnaces. J. FENTON and R. BAIN (B.P. 296,255, 24.1.28).—The retort is tubular with a horizontal or sloping axis, and preferably tapers expanding from the feed end. It is subjected to a to-and-fro or oscillating rotation about its axis.

B. M. VENABLES.

Retorts. A. E. WHITE. From INTERNAT. BITUMENOIL CORP. (B.P. 295,225, 5.4.27).—The material is progressively heated during its passage through an inclined rotary cylinder. The progressive temperatures are obtained by forming the retort with a double shell, the annular spaces being divided into sections filled with "heat-equalising" (heat-conducting) solid material alternating with insulating zones filled with non-conducting material, e.g., air. The rotary part is surrounded by a fixed casing with transverse walls coinciding with the air-space sections, thus forming a number of zones for external heating, each of which is provided with its own furnace, the products of combustion being used to heat separate stills for fractionating or cracking the condensed vapours evolved from the retort. The retort may be operated under vacuum. B. M. VENABLES.

Heat exchanger. N. R. FORSSBLAD (U.S.P. 1,680,145, 7.8.28. Appl., 27.1.25. Swed., 30.1.24).—The two fluids pass in parallel alternate courses through flat channels formed between metal partitions. Heat-conducting filling material is placed in the channels, and the whole group is compressed from the two outside partitions so that the pressure is propagated through the filling material, which thus makes good conductive contact with the partitions. B. M. VENABLES.

Heat transfer. J. E. BELL, Assr. to FOSTER WHEELER CORP. (U.S.P. 1,681,926, 28.8.28. Appl., 17.9.23).—Heat is transferred from a hotter fluid to a cooler fluid by means of a heat-transfer medium or third fluid which is divided into a number of fine streams in contact with each of the first two fluids and circulated between them. B. M. VENABLES.

Heat-exchanging structure for air heaters and the like. O. STRACK, Assr. to PFÄLZISCHE CHAMOTTE- u. THON-WERKE SCHIFFER & KIRCHER A.-G. (U.S.P. 1,679,993, 7.8.28. Appl., 21.3.27. Ger., 23.3.26).—A heat exchanger is constructed of superposed refractory bodies, all of the same external shape and having vertical passages of approximately equal aggregate cross-section, but with one or only a few passages in the

uppermost body and the number of passages increasing in each body downwards with a corresponding decrease in thickness of refractory material and increase in number of passages. The distribution of passages in the blocks is regular. B. M. VENABLES.

Grinding or crushing machines. J. H. PULLEN, LTD., and J. STEWART (B.P. 295,117, 16.7.27).—In grinding mills of the type where a roller runs against a fixed grinding block, a scraper is provided which moves axially to and fro and removes solids accumulated behind the block. The reciprocation is effected by means of a rotating shaft cut with right- and left-hand threads with a nut adapted to engage with each thread in turn. B. M. VENABLES.

Crushing apparatus. S. F. OSSING, Assr. to JEFFREY MANUF. Co. (U.S.P. 1,681,779, 21.8.28. Appl., 26.10.25).—A crushing rotor is constructed of a number of spaced discs on a shaft, with radially projecting arms between the discs. The arms are arranged in line to form groups, each group extending half the axial length; the groups at each end are staggered in relation to each other and are equally spaced circumferentially. B. M. VENABLES.

Pulveriser. H. G. LYKKEN (U.S.P. 1,680,894, 14.8.28. Appl., 16.8.24).—The crushing action takes place between a stationary plate and a rotary member formed with teeth having tangential and radial faces. B. M. VENABLES.

Apparatus for grinding or comminuting rubber and other materials. C. E. GARDNER (B.P. 295,435, 12.5.27. Addn. to B.P. 272,572; B., 1927, 689).—Additional feeding devices such as spiked rollers are used. B. M. VENABLES.

Handling of dusty materials. O. SODERLUND, T. GRAM, and TECHNO-CHEMICAL LABORATORIES, LTD. (B.P. 295,233, 4.5.27).—A small proportion of hydrocarbon liquid, preferably non-volatile, is added at a suitable stage and at a suitable temperature to materials that are or would become dusty. In the case of the drying of fine coal, 3% (on the dry weight) of coal tar is mixed with the wet coal. In the case of air-borne dry materials the binder may be added to the stream before or after passing through the cyclone or other collector for the comparatively coarse material. Or the binder may be mixed with already collected dust and the mixture added to the main stream for recirculation, thus avoiding any harmful effects of direct addition of binder etc. B. M. VENABLES.

Tunnel dryer. M. C. H. O. LECOCQ (B.P. 282,663, 15.12.27. Fr., 22.12.26).—In a tunnel dryer two air circuits are provided, each with a propelling device and heater, passing through separate parts of the tunnel and in opposite directions; one circuit thereby receives fresh air and passes it on the second circuit, which exhausts the moist air. By adjustment of the fans the pressure at both ends of the tunnel may be made to approach atmospheric if desired. Modifications claimed are: the direction of one current is reversed, the effect then being of one extended air circuit; a single fan may be used for the two circuits; the whole apparatus may be contained in rotary cylinders; the tunnel may be annular in shape, the goods and air streams entering

and leaving by circumferential openings and the heaters being placed in the central part of the annulus.

B. M. VENABLES.

Drying granular and pulverulent materials. F. S. TUCKETT (B.P. 295,476, 23.6.27).—The apparatus comprises a number of superposed rotary tables to the uppermost of which the material is fed; it is then spread and scraped off by fixed but adjustable spreaders and scrapers so that it falls to the next table through a central opening, and from that table is scraped at the periphery and so on down the series. The tables are contained within an inner casing, and through the annular space between that and an outer casing the drying gases are first passed in a downward, preferably spiral, direction, then upwards in contact with and counter-current to the material. A proportion of the drying medium may be passed directly to the inner chamber and thermostatic control provided. A magnetic separator may be provided for tramp iron. B. M. VENABLES.

Process and apparatus for [leaching and] washing materials. E. SILVANO and V. L. CERRI (B.P. 282,446, 19.12.27. Italy, 17.12.26).—Previously pulverised or granulated material is sandwiched between two conveyor belts, and the solvent or washing liquid may be passed through the thickness of the material (the belts being permeable) several times in opposite directions, or the solvent may be forced in at one end of the ribbon and flow countercurrent to the direction of motion of the belts and material so that the out-flowing solvent is saturated as far as possible. The belts may be supported on a rigid surface preferably comprising one or more cylinders, which in the case of permeable belts are perforated. Methods of collecting the washed material and of washing the belts ready for re-use are described. The edges of the belts are thickened, giving them a channel shape. B. M. VENABLES.

Method and apparatus for classifying materials. ERZ- U. KOHLE-FLotation GES.M.B.H. (B.P. 287,499, 6.12.27. Ger., 22.3.27).—The material while travelling under water is separated by "cross-currents" (upward jets) of water into coarse and fine grains. The fine grains are further separated by hindered settling in a portion of the apparatus screened off from the cross-currents. B. M. VENABLES.

Centrifugal machines. A. E. WHITE. From LAUGHLIN FILTER CORP. (B.P. 295,916, 20.3.28).—A rotary drum with the cylindrical wall comprising the filtering surface is driven at centrifugal speed. Within the drum is a spiral scraper driven at a slightly different speed. The working edge of the scraper is formed of many small segments inserted in a groove in the spiral and loosely secured by pins through slotted holes, so that under the centrifugal force the segments will always make contact with the filtering wall and keep it clear of residue, which is continuously discharged at one end of the drum. B. M. VENABLES.

Centrifugal machines. T. BROADBENT & SONS, LTD., and B. L. BROADBENT (B.P. 296,200, 13.9.27).—A very simple form of centrifuge comprising a bowl or basket above and A.C. motor below, the whole machine being slung at three points. B. M. VENABLES.

Centrifugal separator. D. T. SHARPLES, Assr. to SHARPLES SEPARATOR Co. (U.S.P. 1,679,790, 7.8.28. Appl., 21.3.25).—The drag bush at the lower end of a centrifugal bowl is formed of laminated phenolic-condensation material, and is frictionally held in place by pressing the feed tube upwards. B. M. VENABLES.

Centrifugal separator. E. KOPKE (U.S.P. 1,679,829, 7.8.28. Appl., 30.11.25. Renewed 23.12.27).—A centrifugal bowl has the wall joined to the bottom by an inclined surface, and within is placed an annular baffle of which the height is adjustable, so that the space between the baffle and the inclined surface is variable. B. M. VENABLES.

Centrifugal liquid purifiers. PRESTON STREET COMBING Co., LTD., and J. W. ADAMS (B.P. 296,178, 27.7.27).—In a centrifugal separator for two liquids, e.g., suint and grease in wool washing, which contain also solid matter that would necessitate frequent cleaning of a de Laval type separator, the bowl is bulged outwardly and jets are situated at the largest circumference, of which the apertures are large enough to discharge the solid matter; a quantity of water is added to the feed equal to that discharged through the jets. In the case of wool washing the number of jets (rather than the size of each jet) is adjusted to discharge suint liquor equal to the diluent water necessary to keep the suint liquor at working strength. B. M. VENABLES.

Clarifier and thickener. H. G. SCHWARZ (U.S.P. 1,682,256, 28.8.28. Appl., 23.3.26).—A vertical shell is formed as a conical settling chamber at the lower part with outlet for settled pulp, and is divided by a partition into upper and lower parts, from which partition is suspended a hood surrounding a central feed opening through the partition. The hood is spaced from the wall of the vessel so that clear liquid can rise to the underside of the partition, whence it is drawn off; a weir and draw off is also provided above the partition for floating matter. B. M. VENABLES.

Filter. E. J. SWEETLAND (U.S.P. 1,680,029, 7.8.28. Appl., 10.1.22).—In a filter that operates within a casing, the filter unit is in the upper part of the casing and a settling zone is formed in the lower part, which is provided with a drain plug and attached scraping or dredging means for removal of heavier impurities. B. M. VENABLES.

Separation, by distillation, of miscible liquids. T. E. PERKS (B.P. 289,394, 16.2.28. N. Zealand, 26.4.27).—The mixture of liquids flows in a continuous course and its temperature is progressively raised. A collecting hood for vapours is placed over the liquid divided by partitions into separate zones for the various fractions, each having its own condenser, which in one form of apparatus may take the form of trays to catch liquid condensed on the water-cooled partitions. The liquid may flow in a zig-zag, spiral, or concentric path. B. M. VENABLES.

Heating or cooling liquids or fluids. ROYLES, LTD., and A. G. ROYLE (B.P. 295,245, 6.5.27).—A heat exchanger is formed of two concentric tubes with end caps affording inlet and outlet connexions for the two fluids. The annular space is formed into a spiral passage by means of a spiral rod equal in diameter to

the width of the annular space and inserted therein. The rod is held in place, and good heat conductivity obtained, by tinning. B. M. VENABLES.

Spraying tower for cooling and crystallising solutions. GEWERKSCHAFT BURBACH, and F. KILLEWALD (G.P. 446,087, 25.7.24).—The apparatus comprises a central feed column through which the hot liquid is passed in a series of interconnected pipes to allow of stage cooling, and from the top of which the solution is sprayed into a number of cooling chambers with inclined floors arranged round the central shaft. A. R. POWELL.

Dehydrating apparatus [for fluids]. W. D. CALLAN, Assr. to ECONOMIC POWDERED PRODUCTS Co. (U.S.P. 1,682,596, 28.8.28. Appl., 5.5.20. Renewed 5.7.27).—The fluid is sprayed into currents of heated air in an upper compartment, and the air passes from the lower part of the upper compartment to the upper part of a lower compartment through a restricted horizontal passage, below which is situated collecting means such as conveyor belts for the powder. After passing through the main collecting zone the air may be exhausted through screens. B. M. VENABLES.

Separation or recovery of a gas or vapour from a mixture of gases or vapours. SILICA GEL CORP., Assees. of F. H. WAGNER, JUN. (B.P. 273,261, 25.5.27. U.S., 24.6.26).—Finely-divided solid absorbent is suspended in a stream of non-absorbable gas—preferably a minor quantity of gas already treated—and sprayed into the main stream of gas to be treated in such a manner that it (the solid) falls by gravity through the gas, the latter preferably following a rising path. The process may be made cyclic and continuous by the use of two cyclone or other separators, between which the solid absorbent is regenerated in a stream of heated gas. B. M. VENABLES.

Separation and recovery of gases and vapours by absorbent solids. A. GODEL (B.P. 295,504, 25.7.27. Addn. to B.P. 267,369; B., 1927, 689).—The apparatus works on the same principle as the subject of the original patent, but the heating tubes are differently arranged, and a series of separated masses of absorbent may be used. B. M. VENABLES.

Adsorption processes [for gases] and apparatus. S. PILAT (B.P. 295,874, 22.11.27).—The container for the absorbent is divided so that in a first charging operation the gases may be passed through the full height of the absorbent mass, and in a second charging operation through only a portion of the mass (or *vice versa*), the second operation always being in the reverse direction to the first; by this means the whole mass is approximately uniformly charged. The treatment of gas may be continuous by having a series of containers to allow for regeneration. B. M. VENABLES.

Separation of dust from a gas or mixture of gases. E. PRAT (B.P. 288,190, 2.4.28. Fr., 1.4.27).—A fan is provided with a volute exceeding 360°, and a minor branch is taken off tangentially and leads to a cyclone collector for the dust. Suction is produced at the outlet of cyclone either directly or indirectly by the same fan; several methods are described for effecting this. B. M. VENABLES.

Separation of gases from mixtures thereof. URBAIN CORP. (B.P. 270,287, 23.4.27. Fr., 1.5.26).—A jet of vapour containing very fine liquid or solid particles, preferably of the same substance as the vapour, is passed through the gas. This jet will remove a greater proportion of the gas constituent which has the greater speed of diffusion and absorption. Means are provided for regulating the speed and pressure of the gaseous mixture and of the jet, also the nature of the latter, the temperature, and size of the conduits.

B. M. VENABLES.

Air- and gas-washing apparatus. E. M. BASSLER (B.P. 296,159, 28.6.27).—The gas enters the apparatus downwardly upon the upper surface of a horizontal, rapidly rotating disc on the centre of which a jet of water or other washing liquid impinges. The gas and spray leave the disc through a restricted annular space between the disc and the top of the apparatus, which space is swept by pins upstanding from the edge of the disc. The spray is caught on a cylindrical depending baffle, and the water is drawn off from the bottom of the main casing while the gas is deflected upwards, and leaves through an outlet which is situated higher than the lower edge of the baffle. [Stat. ref.]

B. M. VENABLES.

Effecting an intimate contact between gases and liquid which have to react on each other. H. PETERSEN (B.P. 295,856, 10.10.27).—A propeller is situated near, and sweeps over the whole surface of, one wall of a reaction chamber, and causes a strong whirling action without causing any admission or exhaust of the gases. To one side of the propeller may be attached a spraying device for the liquid in the form of a hollow stepped pyramid with apertures for exit of the liquid.

B. M. VENABLES.

Determining the temperature of gases. KAISER-WILHELM-INST. F. EISENFORSCHUNG E. V. (B.P. 274,440, 21.6.27. Ger., 16.7.26).—A sample of the gas is continuously aspirated over a thermocouple. To compensate for errors due to cooling of the gases the thermometer is heated by electric resistance elements, such an amount of heat being supplied that no change of temperature is indicated when the rate of aspiration of the gas is suddenly changed.

B. M. VENABLES.

Purification of gases by high-tension discharge. A. WEIGAND (G.P. 446,862, 23.9.24).—The gas stream is passed downwards through permeable brush electrodes, whereby the greater part of the impurities is precipitated, into a chamber the diameter of which increases towards the bottom, from which it rises through a gradually decreasing volume of crude gas to the outlet pipe, traversing an electrostatic field throughout. The electrodes consist of iron gratings covered with sheet iron with large perforations in the middle only.

A. R. POWELL.

Centrifugal separating machine. K. J. SVENSSON and K. A. P. NORLING (U.S.P. 1,683,424, 4.9.28. Appl., 7.1.27. Swed., 10.9.26).—See B.P. 264,777; B., 1927, 241.

Bricks or blocks for furnace building etc. R. HICKTON, W. W. BAGGULEY, and R. G. HICKTON (B.P. 296,033, 18.2.27.)

Gas burners for furnaces. F. LILGE (B.P. 296,204, 22.9.27).

[Differential expansion] apparatus for measuring or controlling temperatures. J. JERIKE (B.P. 268,812, 31.3.27. Ger., 3.4.26).

[Reinforcing or uniting means for] lead columns, pipes, etc. [subject to corrosive action]. Soc. BELGE DE L'AZOTE (B.P. 290,664, 8.5.28. Belg., 20.5.27).

Refrigerating apparatus of the absorption type. G. MAIURI and R. F. BOSSINI (B.P. 296,109 and 296,110, 25.2.27).

Refrigerating apparatus of the absorption type. H. D. FITZPATRICK. From S. K. D. M. VAN LIER (B.P. 296,478, 21.12.27).

Absorption refrigerating apparatus. ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 271,513, 21.5.27. Swed., 22.5.26).

Heat-producing composition (U.S.P. 1,679,432).—See VII. **Rotary drum furnace** (B.P. 284,731).—See X. **Separation of materials** (U.S.P. 1,679,739 and 1,679,740).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Microbiology of coal and its associated strata. II. Microflora of bituminous coal deposits. R. LIESKE and E. HOFMANN (Brennstoff-Chem., 1928, 9, 282—285; cf. B., 1928, 554).—The micro-organisms in a number of Ruhr mines at depths of 400—750 m. have been studied. The mine water was seldom sterile. Iron bacteria (*Gallionella*) and sulphur bacteria (*Thiothrix*) were found in some galleries. Bacteria, chiefly of the *Subtilis* and *Mesentericus* groups, together with various cocci, were found in the coal seams themselves. None of these was pathogenic.

A. B. MANNING.

Hydrogenation of an eocene brown coal under pressure. J. VARGA (Brennstoff-Chem., 1928, 9, 277—282).—An eocene brown coal (moisture 1.1%, ash 7.4%) has been hydrogenated, without the addition of tar, in a 3.7 litre bomb at 450—480°, and under an initial pressure of 75—125 atm. The rate of heating was such as to reach the reaction temperature in 72—77 min., and the bomb was then maintained at this temperature for 1 min., 1 hr., or 3 hrs. On account of the high sulphur content of the coal (3.98%) it was necessary to add 15% of iron oxide in order to obtain a gas free from hydrogen sulphide and an oil with a minimum sulphur content (0.22—0.29%). The iron oxide had also a positive catalytic effect on the process, lowering the temperature of hydrogenation and increasing the yield of oil. From 16.8 to 57.9% of the coal was converted into oil, the best yields being obtained with only a short heating period (1 min.) after reaching the reaction temperature. On further heating the oil was more or less (up to 50%) decomposed again. The maximum conversion into oil (57.9%) was obtained with a charge of 300 g. of coal at 470° and an initial pressure of 100 atm. The yield fell off when the charge was increased. The experiments carried out under an initial

pressure of 125 atm. throw most light on the mechanism of the process. The coal is first converted into high-boiling products, and these subsequently into lighter lower-boiling oils. If decomposition occurs the higher-boiling primary products are first affected. The relative quantity of hydrogen used, the temperature, and the time of heating affect the quality of the oil produced as well as the yield. A. B. MANNING.

Hydrogenation of Japanese coals. Y. OSHIMA and S. TASHIRO (J. Fuel Soc. Japan, 1928, 7, 70—73).—A number of Japanese coals have been hydrogenated in an electrically heated nickel-chromium steel autoclave. The total yield of oil varied from 67 to 96% (calc. on pure coal) according to the coal used and the conditions of temperature and pressure under which the hydrogenation was carried out. The temperature-pressure curves show two characteristic points, one at which the temperature tends to fall in spite of the continued heating, whilst at the other the pressure does not change in spite of the rising temperature. By varying the temperature and pressure from those giving the maximum oil yield it may be possible to increase the yield of the more valuable lighter oils and so render the process more successful economically, although the total oil yield is smaller. A. B. MANNING.

Spontaneous electrification in dust clouds (with special reference to coal dust clouds). S. C. BLACKTIN (Safety in Mines Res. Bd. Paper No. 43, 1928, 19 pp.).—The electrical charge generated in a cloud of coal dust raised by a current of air has been measured by passing the cloud through a copper conductor connected to a quadrant electrometer. The coal was ground to pass 200-mesh. The charge increased with the speed of the air current and with the amount of dust dispersed; it also varied considerably with different samples of coal. Potential differences sufficiently high to produce visible sparks were generated. Experiments with lycopodium spores and with rice and potato starches suggested that, other things being equal, the charges generated per unit weight of material increase with decrease in particle size. This conclusion was confirmed by experiments on coals ground for increasing periods of time, and on elutriated samples of American coals differing in fineness between known limits. A. B. MANNING.

Carbon black. I. Volatile constituents. C. R. JOHNSON (Ind. Eng. Chem., 1928, 20, 904—908).—Carbon black shows on combustion the presence of 5—15% of oxygen, hydrogen, and nitrogen. It is far from certain that these gases are simply adsorbed. These volatile constituents are of importance in relation to the reinforcing power of carbon black when vulcanised with rubber. The volatile matter evolved was found to increase with temperature from 400° to 955°, to be less under reduced pressure (perhaps owing to oxidation being less), and to proceed in two stages as regards time. Carbon black which has been heated to 955° is no longer hygroscopic or adsorbent. The ultimate analysis of a sample, the analysis of the gases evolved, and a combustion of the residue were correlated for five varying samples. "Long" blacks suitable for printing ink gave much higher volatile contents and

higher original oxygen contents than rubber blacks. There is evidence that oxygen is present in the elementary state, and that the oxides of carbon found are reaction products. The hydrogen is the last gas to be driven off. For any particular make of carbon black there is a limit beyond which volatile matter adversely affects the tensile strength of rubber. It is not possible for the volatile matter to be a cause of "blow-outs" in solid tyres. It is suggested that the gas acts as a lubricant during mixing with rubber, and that, therefore, samples of very low volatile content sometimes show an excessive development of heat. C. IRWIN.

Complete gasification of coal for towns' gas. M. W. TRAVERS (J.S.C.I., 1928, 47, 203—210, 213—219 T).—The process of manufacture of water-gas from bituminous coal is discussed. It is shown that in a water-gas plant operated with bituminous coal as fuel the coal can be efficiently carbonised if both the blow gas and the water-gas are allowed to pass through it. If the blow gas be diverted, and only the water-gas is passed through the coal, sufficient heat is not carried to it to effect the process of carbonisation. In the process already described the coal is carbonised by passing through it the water-gas generated from the coke, and an additional quantity of mixed coal gas and water-gas, preheated by passing through chequer-work heated by the blow gas. The heat of the blow gas is thus utilised regeneratively for carbonising the coal. The process has been further developed, and a plant is described in which the coal is carbonised and gasified as in the original process, but the gas can also be enriched in a manner analogous to that employed in the carburetted water-gas plant, but so that the enriched gas does not return to the circulation system, and the tar produced in the process of carburetting does not mix with the coal tar produced by the distillation of the coal. The details of working of the whole process are discussed, particularly with regard to the carbonisation of the coal.

Analysis of gaseous mixtures containing carbon dioxide, carbon monoxide, hydrogen, and methane. W. E. J. BROOM (J.S.C.I., 1928, 47, 276—278 T).—A method for the determination of the above gases by the well-known oxidation process, using copper oxide at 290—300°, is described. The apparatus used is easily constructed from soft soda-lime glass, and the method allows for the differences in molar volume of different gases. Tests show that results accurate to 0.1% on a volume of 10 c.c. may be obtained.

Catalysts used in the synthesis of higher hydrocarbons from water-gas. A. ERDELY and A. W. NASH (J.S.C.I., 1928, 47, 219—223 T).—A series of experiments were carried out with the catalysts cobalt-copper-aluminium oxide (zinc oxide and cerium oxide) and also with an unpromoted cobalt-copper mixture, and the effect of various factors on the interaction of carbon monoxide and hydrogen was studied. Such factors were use of catalyst support, temperature, relative proportion of the two gases, and gas velocity. The use of alumina in catalyst mixtures was found to favour the formation of water rather than of oil; a reverse effect was observed when ceria was the promoter. Temperatures of 280—290° were the most favourable.

When silica gel was used as the catalyst supporting material, only one case was noted where the activity reached that of unsupported catalysts.

Possible chemical utilisation of methane, with special reference to natural gas. A. W. NASH and H. M. STANLEY (*Fuel*, 1928, 7, 397—401).—A summary of the methods which have been proposed for the utilisation of methane, *e.g.*, by chlorination, oxidation to formaldehyde, thermal decomposition with production of carbon black, etc. A. B. MANNING.

Inflammability of automobile exhaust gas. G. W. JONES (*Ind. Eng. Chem.*, 1928, 20, 901—903).—A number of analyses of automobile exhaust gas are given showing inerts varying from 76.9 to 98.5% on the air-free analysis (the oxygen content in the original did not exceed 1.1%). The combustibles are hydrogen, carbon monoxide, and methane. The limits of inflammability for mixtures of these gases singly with either carbon dioxide or nitrogen were determined and depicted graphically. Then for a given exhaust gas the inerts are distributed arbitrarily amongst the combustibles, and the inflammability limits of the mixture may be calculated in accordance with Le Chatelier's law. These results were in good accord with those given by experiment. Exhaust gases are non-inflammable if the air-fuel ratio exceeds 11.75 to 1 by weight. Normal carburettor adjustments are in excess of this, but in special cases danger of explosion may arise. C. IRWIN.

Hydrocarbons in a higher fraction of low-temperature tar. K. KURIHARA (*J. Fuel Soc. Japan*, 1928, 7, 61—62).—The presence of naphthalene (0.4—1.0%), 1-methylnaphthalene, and dimethylnaphthalene in the neutral oil fractions boiling above 200° of low-temperature tars from Fushun coals has been established. 2-Methylnaphthalene was not found. In the fraction boiling above 300° anthracene and methylanthracene were identified. The existence of azulene in the fraction 140—150° (20 mm.), indicated by its deep green colour and blue fluorescence, was confirmed by Kremer's method (B., 1923, 424 A). A. B. MANNING.

Hydrogenation of neutral oil of low-temperature tar. S. TASHIRO (*J. Fuel Soc. Japan*, 1928, 7, 67—70).—A neutral oil fraction of Fushun tar, of boiling range about 200° to 350°, has been hydrogenated ("berginised") in a 600 c.c. autoclave under various conditions of temperature and pressure. Above a definite temperature, about 450°, decomposition of the oil occurs with separation of free carbon. Hydrogenation proceeds most favourably just below this temperature; the initial pressure of the hydrogen should be at least 75 atm. It is, however, impossible to hydrogenate the oil completely, the iodine value of the product still amounting to 65% (approx.) of that of the original oil. In the presence of nickel oxide the hydrogenation is much more complete, and it is possible to bring the iodine value down to 12% of the original. Ferric oxide has a slight favourable effect, and the addition of zinc oxide also lowers the iodine value of the product. Copper and magnesium oxides are without action, whilst the addition of alumina appears to accelerate the decomposition of the oil. A. B. MANNING.

Oil-asphalts and tars. A. N. SACHANOV and N. A. VASILEV (*Neft. Choz.*, 1927, 13, 334—339).—Neutral tar, extracted from fuller's earth after removal of asphaltenes and acid sludge, was unstable, and had a composition corresponding with $C_nH_{2n-m}O_p$, where n is 19—55, m is 9—33, and p is 2—3; these compounds are polycyclic and contain not more than one double linking. They are soluble in sulphuric acid, and may form compounds with it. CHEMICAL ABSTRACTS.

Manufacture of asphalt. A. N. SACHANOV and L. G. SHERDEVA (*Neft. Choz.*, 1928, 14, 513—518).—The possibility of using certain Russian acid sludges is examined. CHEMICAL ABSTRACTS.

Retort construction and the constitution of low-temperature gas benzene. Y. BAN (*J. Fuel Soc. Japan*, 1928, 7, 62—67).—The low-temperature carbonisation plant of the Imperial Fuel Research Institute of Japan consists of six cast-iron vertical retorts with a total throughput of 6 tons per day. Each retort is 11 ft. high and slightly tapered, the section at the centre being 5 in. \times 2 ft. 6 in. The retorts are covered with carborundum brick to prevent overheating, and are heated by horizontal flues. The flue temperature is maintained at about 600°, the temperature of the charge being about 550°. The semi-coke is discharged continuously into water and is then conveyed to a bin. The yields of products from Takasaka and Okinoyama coals were respectively: semi-coke 61.50 and 59.77%, tar 12.20 and 7.78%, gas benzene 0.29 and 0.26%, gas 12.42 and 9.76%. The gas benzenes contained 7—10% of unsaturated hydrocarbons (by loss on washing with 80% sulphuric acid) and 22—25% of aromatics (by the aniline point method). The presence of benzene, toluene, and xylene, indicated by the position of the maxima on the sp. gr. and refractive index curves of the fractionated gas benzene, was confirmed by the isolation of their nitro-compounds. The presence of benzene in the gas benzene is ascribed to local overheating of the retort. A. B. MANNING.

Low-temperature tar oil as a Diesel engine fuel. Y. BAN and T. SUWA (*J. Fuel Soc. Japan*, 1928, 7, 57—60; cf. preceding abstract).—The suitability of low-temperature tar oils from Sakhalin and Fushun coals as fuels for a 33-b.h.p. Diesel engine has been examined. The acid-free tar oils, whether free from pitch or not, gave a slightly better performance than a Diesel oil of petroleum origin. The pitch-free oils, containing tar acids, required a higher fuel consumption, and under light loads gave rise to misfires and detonations. No trouble was experienced with a tar oil containing 1.1% of water. It is concluded that in order to produce a satisfactory Diesel oil from low-temperature tar it is necessary to remove the tar acids, the presence of which raises the auto-ignition temperature and lowers the calorific value. It is also desirable to remove the solid paraffins, which give the oils a high viscosity at ordinary temperatures. A. B. MANNING.

Auto-ignition temperature of Diesel oil. T. SUWA (*J. Fuel Soc. Japan*, 1928, 7, 53—56).—The auto-ignition temperatures of a number of low-temperature tar oils have been determined in an improved Moore

type apparatus provided with a platinum crucible of 14.8 c.c. capacity. The determinations were made in an atmosphere of oxygen, supplied at the rate of 15 c.c./min. Low-temperature tar and the pitch-free tar oils gave higher values (e.g., 337° and 405°, respectively, for the tar from Fushun coal) than a Diesel oil of petroleum origin (about 260°); the acid-free oils gave values below 300°. Varying the rate of passage of oxygen from 10 to 30 c.c./min. had no effect on the behaviour of the petroleum and the neutral tar oils, but with oils containing tar acids the auto-ignition temperatures varied with the rate of oxygen supply. The auto-ignition temperatures of a series of neutral oil fractions varied only slightly with their b.p.

A. B. MANNING.

Comparison of Diesel fuel oils from Baku, Grozni, and Emba. N. TSCHERNOSHIKOV (Nef. Choz., 1927, 13, 323—327).—The characteristics of the above oils are recorded.

CHEMICAL ABSTRACTS.

Sakhalin crude oils. S. S. NAMETKIN and V. G. PUZILLO (Nef. Choz., 1928, 14, 519—520).—Data concerning crude oils from the Okha oil field, and distillates obtained therefrom, are recorded.

CHEMICAL ABSTRACTS.

Use of lubricating greases for preservation of metals. M. V. BORODULIN (Trans. State Inst. Appl. Chem., Moscow, 1927, [5], 53—58).—Adhesiveness is more important than viscosity, but the only measure available of the former is a determination of the latter. The presence of free naphthenic acids is deleterious, but the addition of soap to neutralise the effect of free acids tends to facilitate the action of atmospheric oxygen on the metals.

CHEMICAL ABSTRACTS.

Carius determination of sulphur in less volatile petroleum oils. J. M. DEVINE and F. W. LANE (J. Amer. Chem. Soc., 1928, 50, 1707—1710).—The following modification gives accurate results with oils containing more than 0.5% S and also with *n*-butyl sulphone. Approximately 0.25 g. of oil and 4 c.c. of fuming nitric acid are heated during three 7-hr. periods to 175°, from 175° to 225°, and from 225° to 300°, the gases being released after each heating. The analysis is completed as usual.

H. E. F. NOTTON.

Determination of unsaturateds in lubricating oils. F. S. BACON (Ind. Eng. Chem., 1928, 20, 970—971).—The sample (1—10 c.c.) diluted with at least 10 c.c. of benzene or gasoline of known unsaturated content, is added to 25 c.c. of 10% sulphuric acid. Small quantities of 0.5*N*-sodium or -potassium bromide-bromate solution are added with vigorous shaking until the change in colour indicates an excess of free bromine. Saturated potassium iodide solution (1—2 c.c.) is next added. If a brown colour is not developed the test is repeated using a greater volume of bromide-bromate solution; otherwise, the mixture is back-titrated using 0.2*N*-sodium thiosulphate to the disappearance of the brown colour, and an excess of 1—2 c.c. of thiosulphate is added. The oil and water layers are now separated, the oil layer is washed, and starch indicator added to the combined aqueous solution, which is then titrated to a blue coloration using 0.1*N*-iodine (cf. Francis, B., 1926, 811).

W. S. NORRIS.

Adsorption by bone char. WAYNE.—See XVII.

PATENTS.

Carbonising apparatus. H. HENNEBUTTE and E. GOUTAL (U.S.P. 1,680,613, 14.8.28. Appl., 28.12.23. Fr., 3.1.23).—An annular drying or carbonising chamber is formed between two vertical metal cylinders, arranged concentrically one within the other, and enclosed within a brick setting. The lower end of the chamber is closed by an inturned flange on the outer cylinder, but the upper end is open and in communication with an annular flue surrounding the chamber. Hot gases can pass from this flue, down through the material in the chamber, and out through perforations in the lower part of the inner cylinder.

A. B. MANNING.

Carbonisation plant. CHAMBER OVENS, LTD. From PINTSCH & DR. OTTO GES.M.B.H. (B.P. 295,885, 7.12.27).—The end walls of retort oven benches etc. are constructed of a number of juxtaposed, curved, wall segments, which ensure uniform distribution of the stresses due to expansion when the setting is heated. Buckstays are interposed between the adjacent segments, which are held in contact with the brickwork walls of the plant by means of resiliently mounted ties.

A. B. MANNING.

[Low-temperature] carbonising or like retorts. C. TURNER (B.P. 295,461, 31.5.27).—In the lower part of a vertical retort are two or more, preferably five, intermeshing Archimedean screws on vertical axes, which support the charge and by their rotation regulate its rate of discharge. The conditions of carbonisation and the pitch and dimensions of the screws may be such that the discharge of the coke from the retort occurs without rotation of the screws. Passing up centrally through the screws is a steam inlet pipe. The offtake of the retort contains a valve which is opened at intervals to permit rapid escape of the volatile products of carbonisation, and is closed again when the pressure within the retort has fallen to the desired limit.

A. B. MANNING.

Means for distilling carbonaceous material. J. H. ANDERSON (B.P. 295,503, 25.7.27).—The drum of a horizontal rotary retort for the low-temperature carbonisation of carbonaceous materials is provided with inwardly projecting annular flanges at intervals in order to prevent lateral movement of loose heavy masses of metal, which are placed within the drum to assist in the transfer of heat and to prevent the formation of a heat-insulating coating. The discharge end of the drum, which is entirely open, projects into a box containing a conveyor for the removal of the carbonised material. A pipe communicating with this box carries off the distillation gases. The drum projects beyond the ends of the combustion chamber in which it rotates, the escape of the products of combustion being prevented by annular flexible diaphragms attached to the drum and making contact with seatings on the end of the combustion chamber. The hot combustion gases from suitably placed gas jets below the drum pass round the latter and escape through flues arranged alternately with the inlet flues for the combustion air, which thereby becomes preheated.

A. B. MANNING.

Construction of gas and coke ovens. F. TOTZEK (B.P. 295,906, 16.2.28).—Coke oven walls with vertical flues are built up of courses in which stretchers are interspersed with bricks having symmetrically arranged limbs projecting towards the interior of the hollow wall; these limbs are either joined to the bricks forming the transverse walls or are of such length as to extend across the cavity, forming the transverse walls themselves.

A. B. MANNING.

Distillation of solid carbonaceous materials and retorts therefor. H. NIELSEN and B. LAING (B.P. 295,755, 18.5.27. Cf. B.P. 287,381; B., 1928, 395).—A rotary and preferably inclined retort is fitted with sets of shelves or baffles, in the form of truncated pyramids, the narrower ends of which are directed towards the discharge end of the retort. As the retort rotates, the material to be carbonised is carried upwards into the zones through which the heating gases are passed, and is heated, not only by the hot gases travelling between the periphery of the retort and the exterior of the shelves, but also by heat transmitted through the shelves from the gases passing beneath them.

A. B. MANNING.

Continuous distillation of carbonaceous fuel. W. E. TRENT, Assr. to TRENT PROCESS CORP. (U.S.P. 1,675,315, 26.6.28. Appl., 22.12.22).—Coal or a mixture of coal and oil is distilled in a series of containers through which hot gases are passed; successive containers being thus subjected to progressively lower temperatures, different oil fractions distil from one container to the next or are collected in traps placed between the containers. As coking is completed in one container the maximum temperature is transferred to the next.

C. HOLLINS.

Carbonaceous fuel and its manufacture. A. OBERLE, Assr. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,674,837, 26.6.28. Appl., 9.5.23. Renewed 13.1.28).—Residues from cracked petroleum are treated at 170–340° with steam under 10 lb. pressure, and the non-volatile matter (which now contains active carbon) is dried under reduced pressure and treated with gaseous cracking products under pressure.

C. HOLLINS.

Low-temperature carbonisation. KOHLENVEREDLUNG A.-G., Assecs. of KOHLENVEREDLUNG GES.M.B.H. (B.P. 267,505, 7.3.27. Ger., 9.3.26).—Illuminating or heating gas is produced by the addition of water-gas or generator gas to low-temperature gases from externally-heated retorts, the added gas being fed through or from one side to the other of the charge as it moves in a thin layer over the heating surfaces. The added gas may be preheated by the waste gases, hot coke, or other means. In retorts provided with means for the fractional distillation of carbonaceous materials, the added gas is fed separately in suitable amounts to the different gaseous fractions.

A. B. MANNING.

Combustion with the aid of a catalyst. T. W. GRUETTER (B.P. 276,300, 10.3.27. U.S., 23.8.26).—An oxide of copper, cobalt, or nickel, or a mixture of these, supported, if desired, on a suitable carrier, is used as a catalyst for the flameless combustion of fuels for industrial and domestic heating purposes. To initiate com-

bustion, the catalyst requires heating to only a moderate temperature, well below that of incandescence.

A. B. MANNING.

Utilising waste heat in gas-producing plant. FRANKFURTER GAS GES., and F. REICHARD (B.P. 295,900, 31.1.28).—The blow gases of a water-gas plant are used to heat a waste-heat boiler which is also heated by auxiliary fuel; the supply of the latter is throttled down during the blow period to such an extent that the total heat supplied to the boiler per unit time is maintained constant.

A. B. MANNING.

Purification of gases. W. H. FULWEILER, Assr. to U.G.I. CONTRACTING CO. (U.S.P. 1,679,858, 7.8.28. Appl., 4.12.23).—In the purification of gases by absorption of the hydrogen sulphide in an alkaline salt solution, the foul solution is revived by being passed in a thin film over coke in the presence of air. The sulphides present are thereby oxidised to sulphur and the original salt is regenerated.

A. B. MANNING.

Drying of fuel gases. C. COOPER, D. M. HENSHAW, and W. C. HOLMES & Co., LTD. (B.P. 295,411, 7.4.27).—In carrying out the processes of B.P. 248,841, 285,936, and 287,678 (B., 1926, 428; 1928, 325, 396) the rate of circulation of the liquor, which is maintained constant, and the total volume of liquor are so chosen that temporary, *e.g.*, diurnal, variations in the gas consumption produce only negligible variations in the average concentration of the liquor and in the dryness of the treated gas. In order to meet seasonal variations in the gas consumption, the area of heating surface with which the liquor is brought into contact for reconcentration is correspondingly varied.

A. B. MANNING.

Recovery of carburetted gases as fuel [alcohol] from the distillation of natural or waste products. E. CLAUSEN (F.P. 625,379, 20.9.26).—The residues from oil distillation, waste wood, peat, or coal slack are subjected to destructive distillation, and the gases evolved are passed through a scrubber and thence into sulphuric acid in the presence of a catalyst to absorb the unsaturated hydrocarbons. The acid is then diluted and distilled with steam to hydrolyse the alkyl sulphonates with the production of a crude alcohol for use as motor fuel.

A. R. POWELL.

Recovering acetylene in a concentrated form from gaseous mixtures thereof with inert or reducing gases free from oxygen or oxygen-containing compounds. I. G. FARBERIND. A.-G. (B.P. 280,885, 28.10.27. Ger., 16.11.26).—The gases are washed with water under pressure; the acetylene is dissolved and is recovered from the solution on releasing the pressure.

A. B. MANNING.

Distillation of coal tar. C. WESSEL (B.P. 273,675, 8.6.27. Ger., 29.6.26).—Tar, heated sufficiently to prevent subsequent condensation of steam therein, is subjected to the action of live steam at as low a temperature as possible in order to drive off, in separate stages, the light oil and water, and then substantially pure naphthalene. The steam supply is then discontinued and the residual tar distilled by direct heating.

A. B. MANNING.

Manufacture of [bituminous] emulsions. L. KIRSCHBRAUN (U.S.P. 1,679,475, 7.8.28. Appl., 24.3.26).—A colloidal emulsifying agent is mixed with sufficient of a previously prepared emulsion of similar character to form a paste, heated bituminous material in the liquid state is added, and the whole is agitated.

A. B. MANNING.

Treatment of montan wax. J. Y. JOHNSON. From I. G. FARBERIND. A. G. (B.P. 296,145, 9.6. and 22.12.27).—The tendency of bleached (oxidised) montan wax to crystallise is avoided by esterifying or otherwise converting the carboxyl groups present into groups not containing free hydroxyl. For boot polishes etc. the wax is preferably partly esterified with glycol and the remaining carboxyl groups are neutralised with lime.

C. HOLLINS.

Production of low-b.p. oils by thermal treatment of coals, oils, etc. I. G. FARBERIND. A. G. (B.P. 272,194, 23.5.27. Ger., 1.6.26).—The starting materials are subjected, generally in the liquid state, to heat treatment at 300–500°, under any desired pressure and in the presence of catalysts. The resulting products, in the state of superheated vapour, are passed over other catalysts at a temperature substantially higher than that used in the first stage of the process. Either or both stages may be carried out in the presence of gases, preferably hydrogen.

A. B. MANNING.

Conversion of heavier hydrocarbons into lighter hydrocarbons. R. CROSS (B.P. 273,256, 12.5.27. U.S., 28.6.26. Cf. B., 1923, 260 A; 1925, 951).—Hydrocarbons of the kerosene and gasoline series, having a critical temperature not above 490°, are converted into motor fuel by passage through a heating zone into a large insulated reaction chamber; the charging stock is raised in the heating zone to a temperature above its critical temperature, but passes into the reaction chamber before substantial decomposition takes place. The pressure is maintained at a value (700–1000 lb./in.² or higher) above the critical pressure of most of the hydrocarbons present. The reaction products pass successively through a polymeride separator and a bubble tower to a condenser. The final product is a motor fuel low in unsaturated hydrocarbons, high in naphthenes, and possessing marked anti-detonating qualities.

A. B. MANNING.

Synthesis, distillation, cracking, and hydrogenation of hydrocarbon oils. J. TRAUTMANN (B.P. 261,786, 20.11.26. Ger., 21.11.25).—In carrying out these processes the necessary heat is transmitted to the oil by spraying hot finely-divided metals, in the form of powder, liquid, or vapour, into the reaction chamber. The reaction substances themselves may be used to assist in the atomisation of the metals, or may be sprayed into the chamber simultaneously through adjacent nozzles, so arranged as to give as intimate contact as possible of the substances with the metal. Catalysts in a finely-divided form may, if desired, be sprayed similarly into the reaction chamber.

B. MANNING.

Treatment of gasoline and similar petroleum products. A. G. BLOXAM. From ALLGEM. GES. F. CHEM. IND. M.B.H. (B.P. 295,25 3,7.5.27; cf. B.P. 245,072; B., 1926, 621).—Gasolines having a substantial

proportion of constituents boiling above 160° are improved in volatility and in "anti-knocking" qualities by the removal of the higher-boiling saturated hydrocarbons. The unsaturated hydrocarbons in the fraction of the oil boiling from about 160° to 250° are extracted therefrom by liquid sulphur dioxide, acetone, or mixtures of these, and, after recovery from the extracting agent, are added to the fraction boiling below 160°.

A. B. MANNING.

Purification of liquid hydrocarbons by liquid sulphur dioxide in counter-current. ALLGEM. GES. F. CHEM. IND. M.B.H. (B.P. 279,774, 18.2.27. Ger., 1.11.26).—To ensure complete separation of the liquid sulphur dioxide from the oil in an apparatus in which the latter is continuously washed by a counter-current of the former, the head of the washing tower, or the container into which the oil flows from the tower, is made of greater cross-section than the tower itself, so that the velocity of flow of the oil is reduced and any entrained drops of liquid sulphur dioxide are given time to settle.

A. B. MANNING.

Manufacture of unsaturated hydrocarbons. I. G. FARBERIND. A. G. (B.P. 267,155, 7.3.27. Ger., 8.3.26).—Liquid or solid hydrocarbons are mixed with oxygen, or with gases containing at least 50% of oxygen, in amount insufficient for the complete oxidation of the hydrocarbons, and are subjected to incomplete combustion at 650–900°. Water, in the form of liquid or vapour, may be added if desired. Gases rich in olefines are produced.

A. B. MANNING.

Refining and stabilisation of hydrocarbons. E. A. PRUDHOMME, ASST. to SOC. INTERNAT. DES PROC. PRUDHOMME (S.I.P.P.) (U.S.P. 1,674,796, 26.6.28. Appl., 7.4.26. Fr., 4.3.26).—Vapours of hydrocarbon fractions, b.p. below 400°, are treated above 180° with nickel oxide and copper oxide in succession to remove impurities, and are then passed with hydrogen (etc.) first over a nickel catalyst and finally over active carbon. An oil distilling at 60–220° is thus obtained (cf. B.P. 267,138; B., 1928, 220).

C. HOLLINS.

Oil-refining still. O. E. ANDRUS and S. HERMANSON ASSTs. to A. O. SMITH CORP. (U.S.P. 1,680,276, 14.8.28. Appl., 29.10.27).—A vessel is lined with a sheet of corrosion-resisting alloy which is electrically spot-welded at numerous points to prevent distortion and give good heat conduction. The welding must be done in such a way as not to change the character of the lining.

B. M. VENABLES.

Fuel-dust furnace. G. SZIKLA and A. ROZINEK (U.S.P. 1,680,183, 7.8.28. Appl., 24.1.25. Hungary, 4.2.24).—See B.P. 228,906; B., 1926, 572.

Apparatus for washing coals. P. WOLF (U.S.P. 1,682,820, 4.9.28. Appl., 22.10.23. Fr., 24.10.22).—See B.P. 206,151; B., 1925, 162.

Recovery of petroleum from oil-bearing sands. H. ATKINSON (B.P. 296,213, 10.10.27).—See U.S.P. 1,651,311; B., 1928, 45.

Means for forming combustible mixtures for use in internal-combustion engines. C. N. POGUE and A. J. ANDREWS (B.P. 296,238, 19.12.27).

[Oil burner for] combustion of hydrocarbons. J. L. BREESE, JUN. (B.P. 295,758, 23.5.27).

Retorts (B.P. 295,225 and 296,255).—See I. Oil colours from petroleum pitch (G.P. 447,470).—See XIII.

III.—ORGANIC INTERMEDIATES.

Methyl alcohol from hydrogen and carbon monoxide. R. L. BROWN and A. E. GALLOWAY (Ind. Eng. Chem., 1928, 20, 960—966).—The synthesis of methyl alcohol was studied on a small scale, using a reaction tube and receiver of chrome-vanadium steel plated with copper. Uncombined gas could be recirculated. The gases were freed from oxygen, carbon dioxide, and moisture. Pressure-time curves are given for the following catalysts: Zinc oxide, basic zinc chromate, and normal zinc chromate. The chromic oxide considerably accelerates the reaction at temperatures up to 400°. The methyl alcohol actually recovered amounted to 70—95% of that calculated from the pressure drop. The principal by-product was methane. 1 hr.-tests at 400° and under constant pressure (180 atm.) with various space velocities were then made, the yield with normal zinc chromate being the best. The activity of zinc oxide was found to diminish more than 50% after 5 hrs.' use at 400°; the deterioration of zinc chromate was much more gradual. The best chromate catalysts are prepared by reduction at as low a temperature as possible (300°). The data indicate the probability of equilibrium at 400° and 180 atm. at 20% conversion. This point is investigated theoretically, and a method of calculating conversions under other conditions is indicated. C. IRWIN.

Oxidation of anthracene by oxides of nitrogen. M. A. ILJINSKI and B. V. MAXAROV (J. Chem. Ind., Moscow, 1928, 5, 469—473).—Oxides of nitrogen, whether mixed with air or alone, have no oxidising action on nitrobenzene, or on anthraquinone suspended in the latter. Anthracene in nitrobenzene solution is under these conditions quantitatively oxidised to anthraquinone, and 2-methylantracene to the corresponding methylantranthraquinone, but no further. A series of products is obtained from carbazole, chiefly 3:6-dinitrocarbazole, with an admixture of 3- and 9-nitroso-carbazole, 3-nitrocarbazole, 9-nitroso-3-nitrocarbazole, and 3-nitroso-6-nitrocarbazole. Phenanthrene yields a mixture of tarry products, which were not separated. Anthraquinone cannot be isolated from the reaction products when very impure samples of anthracene are taken. Where 25% anthracene is used, the yield of anthraquinone is only about 40% of the theoretical, and the product is contaminated with nitrogenous and tarry matter, from which about one third of the anthraquinone is inseparable. The anthraquinone so derived from crude anthracene cannot be purified by sulphonation. R. TRUSZKOWSKI.

PATENTS.

Polymerisation and purification of hydrocarbons. S. P. MILLER and J. B. HILL, Assrs. to BARRETT CO. (U.S.P. 1,679,093, 31.7.28. Appl., 5.8.26).—Solvent naphtha, benzol, or other hydrocarbon, mixed with a polymerising agent, is passed continuously through an

emulsifier. The polymerisation is hastened and the quality of the resulting resins is improved.

F. G. CLARKE.

Manufacture of aromatic amines from nitro-compounds. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 295,824, 4.8.27).—Since sodium or ammonium polysulphides and thiosulphates are reduced to sulphides, especially in presence of silica gel, heavy metal oxides, etc., by hydrogen or carbon monoxide at high pressures (100—200 atm.) and temperatures (150—180°), a small amount of sulphide is sufficient in the presence of these gases under these conditions to reduce a large quantity of aromatic nitro-compound. The sulphide may be replaced by caustic alkali (or ammonia) and sulphur, or by polysulphides or thiosulphates. Part of the thio-sulphate formed (or introduced) is converted at the temperatures used into sulphate; by employing 1.7—5% of the ammonium sulphide theoretically required there is obtained at the end of the reduction ammonium sulphate free from sulphide or thiosulphate. The hydrogen and carbon monoxide need not be purified. The reduction of nitrobenzene, *m*-dinitrobenzene, *p*-nitroaniline, and *p*-nitrophenol to amines is described. C. HOLLINS.

Manufacture of 1-methyl-2:5-dichloro-4-amino-benzene [2:5-dichloro-*p*-toluidine]. I. G. FARBENIND. A.-G. (B.P. 287,110, 8.3.28. Ger., 15.3.27).—5-Chloro-*o*-toluidine is nitrated at 0° in mixed acid, the reaction mixture is diluted, diazotised with solid nitrite, and the diazo solution decomposed with cuprous chloride solution at 70° to give 2:5-dichloro-4-nitro-toluene, m.p. 40—45°, which is reduced to 2:5-dichloro-*p*-toluidine, m.p. 90—91°, b.p. 130—133°/12 mm. The yield is 80% on the 5-chloro-*o*-toluidine. C. HOLLINS.

Conversion of cyanonaphthalenesulphonic acids, and products of the conversion. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 296,010, 21.2.27).—Naphthalenesulphonic acids containing a cyano-group *o*- or *p*- to the sulphonic group are converted by caustic alkali or alkaline salts into hydroxy-nitriles, -amines, or -acids, and by alcoholic alkali into corresponding alkoxy-derivatives, at 80—150°. The following compounds are described: 2-cyano- α -naphthol, m.p. 179°; 1-hydroxy-2-naphthoic acid, m.p. 186°; 4-cyano- α -naphthol; 3-hydroxy-6-sulpho-2-naphthoic acid; 3:6-dihydroxy-2-naphthoic acid; 1-hydroxy-4-naphthoic acid, m.p. 184—185°; 2-methoxy-1-naphthamide, m.p. 189°; 1-methoxy-2-naphthamide, m.p. 156—157°; 1-methoxy-2-naphthoic acid, m.p. 127°; 4-methoxy-1-naphthamide, m.p. 237°; 4-methoxy-1-naphthoic acid, m.p. 239°; 4-ethoxy-1-naphthamide, m.p. 244°; 4-ethoxy-1-naphthoic acid, m.p. 214°; 4-*n*-butoxy-1-naphthamide, m.p. 250°; 4-*n*-butoxy-1-naphthoic acid, m.p. 208°. C. HOLLINS.

Manufacture of *N*-dihydro-1:2:1':2'-anthraquinoneazine [indanthrone]. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,106, 22.2.27).—Improved yields of indanthrone are obtained by adding to the alkaline melt of β -aminoanthraquinone (i) sodium formate, acetate, or propionate, together with (ii) salts of butyric or higher aliphatic acids, or alkali alkoxides or phenoxides. [Stat. ref.] C. HOLLINS.

Manufacture of products applicable for making dye preparations and of dye preparations made therewith. SOC. CHEM. IND. IN BASLE (B.P. 271,898, 27.5.27. Switz., 29.5.26).—Still residues from distillation of benzaldehyde are sulphonated with oleum or chlorosulphonic acid, and used as dispersing and drying-down agents for acetate silk dyes and other dyes.

C. HOLLINS.

Preparation of stable emulsions, suspensions, and colloidal dispersions of organic substances insoluble in water. M. BUCHNER and R. UHDE (G.P. 446,162, 16.8.22).—For the preparation of stable emulsions of oils, fats, waxes, resins, and similar insoluble compounds a salt of a highly chlorinated fatty acid or resin acid is used as the emulsifying agent.

A. R. POWELL.

Extraction of butyric acid and its homologues. J. LEFRANC, ASST. to SOC. DES BREVETS ÉTRANGERS LEFRANC & CIE. (U.S.P. 1,683,198, 4.9.28. Appl., 25.1.27. Fr., 27.8.26).—See B.P. 276,617; B., 1928, 280.

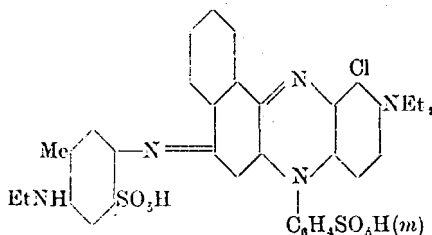
Production of soluble salts of organic compounds possessing acid character. M. BUCHNER, ASST. to A. F. MEYERHOFFER (U.S.P. 1,683,159, 4.9.28. Appl., 22.12.25. Ger., 16.1.25).—See B.P. 246,142; B., 1927, 733.

Conversion of cyanonaphthalenesulphonic acids. R. HERZ, F. SCHULTE, and W. ZERWECK, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,669,297, 8.5.28. Appl., 10.11.27. Ger., 17.5.26).—See B.P. 296,010; preceding.

IV.—DYESTUFFS.

PATENTS.

Manufacture of acid dyes of the phenonaphthasafranine series. J. R. GEIGY A.-G. (B.P. 284,614, 31.1.28. Ger., 31.1.27. Addn. to B.P. 265,986; B., 1928, 441).—Greener dyes are obtained by using in the prior process isorosindulinedi- or tri-sulphonic acids containing halogen in position 1; one sulphonic group is always in the arylimino-group and *ortho* to the imino-nitrogen. Thus the phenonaphthasafraninedisulphonic acid of formula—



is obtained by condensing 6-ethylamino-*m*-toluidine-4-sulphonic acid with the disulphonic acid prepared by the action of bisulphite on 1-chloro-3-diethylisosinduline-12-sulphonic acid (from *m*-sulphophenyl- β -naphthylamine and 3-chloro-4-nitrosodiethylaniline). It gives fast greenish-blue shades on wool.

C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 295,645, 14.5.27).—1:2:6:7-Diphthaloylacridone, from 2-aminoanthraquinone condensed with 1-chloroanthraquinone-2-carboxylic acid and cyclised,

is nitrated with mixed acid at 10–40°, and the product is reduced. The *amine*, which differs from Eckert and Halla's 4-amino-1:2:6:7-diphthaloylacridone (A., 1914, i, 994), is a grey-blue vat dye, and on benzoilation gives a fast olive-green vat dye. The isomeric 3:4:6:7-diphthaloylacridone similarly yields a grey-blue vat dye and on benzoilation a fast violet-grey vat dye.

C. HOLLINS.

Manufacture of vat dyes [of the anthraquinone series]. BRIT. DYESTUFFS CORP., LTD., A. DAVIDSON, and A. SHEPHERDSON (B.P. 295,770, 26.5.27).—A 1-arylaminoanthraquinone-2-carboxylic acid is heated in a high-boiling solvent (trichlorobenzene) with thionyl chloride largely in excess of that required to form the acid chloride. The product, with or without isolation, is then chlorinated at 190°. 1-Anilino- or 1-*p*-toluidinoanthraquinone-2-carboxylic acid gives a pink vat dye, the 1- β -naphthylamino-compound an orange-brown.

C. HOLLINS.

New azo dyes. BRIT. DYESTUFFS CORP., LTD., R. BRIGHTMAN, and P. CHORLEY (B.P. 296,047, 22.4.27. Cf. B.P. 296,011; B., 1928, 782).—Dyes giving level shades on viscose silks, and on wool dyeings fast to milling, are obtained by coupling a tetrazotised 4:4'-diaminodiphenyl mono-, di-, or tri-sulphide with 2 mols. of a 2:8-aminonaphtholsulphonic acid, or with 1 mol. of such acid and 1 mol. of a coupling component of the benzene or naphthalene series. Examples are: 4:4'-diaminodiphenyl sulphide with salicylic acid and phenyl- γ -acid (brown), or with 2 mols. of γ -acid (violet-brown on viscose, maroon on wool); the disulphide with 2 mols. of γ -acid (violet-brown); the trisulphide with salicylic acid and γ -acid (red-brown), or acetyl- γ -acid (yellowish-red).

C. HOLLINS.

Manufacture of acid dyes of the phenonaphthosafranine series. P. LAEUGER, ASST. to J. R. GEIGY SOC. ANON. (U.S.P. 1,683,559, 4.9.28. Appl., 28.12.27. Ger., 13.2.26).—See B.P. 265,986; B., 1928, 441.

Dye preparations (B.P. 271,898).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Plasticity of wool. J. B. SPEAKMAN (Proc. Roy. Soc., 1928, B, 103, 377–396).—The plasticity of wool fibre has been determined under various conditions, *e.g.*, of humidity, by stretching a fibre to a definite extent and measuring the decrease in the tension of the fibre which subsequently takes place. The plasticity increases with increasing humidity, and it is considered that the plasticity is due to the rupture of amide linkages in the wool protein following adsorption of water. Treatment by chemicals, such as acids, which combine with tervalent nitrogen and thereby inhibit addition of the elements of water to the amide linkage decreases the plasticity. The mechanical structure of the elastic and plastic elements in the wool fibre is also discussed.

W. O. KERMACK.

Action of chlorine on jute fibre. H. W. STRONG (J.S.C.I., 1928, 47, 196–198 r).—It is shown that in the interaction between chlorine and jute fibre the simple substitution of chlorine for hydrogen is not the only reaction which occurs, but that it is accompanied by oxidation. The amount of chlorine combined with the

lignone is 5% on the oven-dry fibre sample, and that as hydrochloric acid is 9.9%. The former value corresponds to the formation of a tetrachloro-compound with the lignone.

Behaviour of lignin and chlorolignin in the preparation of wood pulp by means of chlorine.

II. P. WAENTIG (Z. angew. Chem., 1928, 41, 1001—1005; cf. B., 1928, 712).—Chlorolignins produced in the chlorination processes from woody and other fibres have a mean chlorine content of 30% if extracted by organic solvent. In water-extracted chlorolignins the average value is 18—19%, but further extraction of these with organic solvents yields the higher chlorinated product. The most successful extraction of chlorolignin occurs when the preliminary cooking of the fibre with soda is abandoned. Chlorolignin so obtained decomposes slowly if moist and more rapidly by heating, yielding hydrochloric acid. In a sealed tube two thirds of its chlorine is recovered as hydrochloric acid. The amount of soda necessary to dissolve chlorolignin in the cold is less than the chlorine equivalent, but at 95—100° more soda is required. Lime precipitates chlorolignin from soda solution; hence the disadvantages attaching to the use of lime and bleaching powder in processes for cellulose production. The lime-chlorolignin precipitate is easily hydrolysed, and a large excess of lime is required for complete precipitation. The mol. wt. of chlorolignin in acetone is about 1000. A. G. POLLARD.

PATENTS.

Preparation of artificial silk with special mechanical properties.

N. V. NEDERLANDSCHE KUNST-ZIJDEFABR. (B.P. 285,890, 11.11.27. Holl., 25.2.27. Addn. to B.P. 282,721; B., 1928, 706).—The increase in strength of viscose threads may be achieved by leading the spinning bath liquor in counter-flow to the spun material, or by leading the thread over a large number of preferably rotating thread-guides so that it runs in a slightly zig-zag fashion. [Stat. ref.]

D. J. NORMAN.

Manufacture of filaments, threads, bands, etc.

from viscose. COURTAULDS, LTD., and J. H. TAYLOR (B.P. 294,805, 10.10.27).—The emulsification of small quantities of petroleum jelly with viscose solution for the production of viscose threads of subdued lustre (cf. B.P. 273,386; B., 1927, 649) is facilitated if the petroleum jelly is first mixed with 5—10% of its weight of cyclohexanol. Other suitable additions such as 5% of oleic acid may also be made. D. J. NORMAN.

Application of cellulose esters and ethers, and products thereby obtained.

BRIT. CELANESE, LTD. (B.P. 274,841, 1.7.27. U.S., 23.7.26).—Threads, fabrics, films, etc., made of or having a surface of cellulose acetate or other fatty acid esters or ethers of cellulose, are treated with a solution of another or of the same fatty acid ester or ether of cellulose having different solubility characteristics, the solvent employed in the coating solution being a non-solvent for the cellulose compound of the material to be coated. The original surface is thus protected from dampness or friction, any effects therein are rendered permanent, and other effect

materials may be introduced by means of the coating solution. F. R. ENNOS.

Composition for preserving food products.

H. BUEL, Assr. to W. P. HAMMOND (U.S.P. 1,680,934, 14.8.28. Appl., 8.9.21).—A composition for the manufacture of milk containers comprises a mixture of ethylcellulose acetate deposited from acetone solution and a tasteless, odourless, light oil as plasticising agent.

A. R. POWELL.

Making paper pulp. R. W. HOVEY (B.P. 295,759, 23.5.27).—During the cooking of wood under pressure using direct steam, a uniform temperature may be maintained throughout the digester by continuously withdrawing liquor from the top of the digester and injecting it (together with steam, if desired) into the bottom thereof through a series of nozzles so arranged, e.g., annularly, that the hot liquor is forced up through the cooler peripheral zone. A more uniform product is thus obtained, particularly in sulphite cooking.

D. J. NORMAN.

Continuous cooking of fibrous material.

T. L. DUNBAR, Assr. to CHEMIPULP PROCESS, INC. (U.S.P. 1,679,336, 31.7.28. Appl., 25.10.26).—Fibrous material is passed through a series of cooking chambers arranged end to end, and is treated therein with hot cooking liquor. In the first chamber the cooking is effected under hydrostatic pressure, and in succeeding chambers under super-atmospheric pressure at increasing temperatures.

D. J. NORMAN.

Manufacture of straw pulp and paper.

C. H. DEDRICK, Assr. to PHILADELPHIA QUARTZ CO. (U.S.P. 1,682,834, 4.9.28. Appl., 6.1.25).—Straw is cooked with sodium silicate, beaten with the liquor, and sized.

F. G. CROSSE.

Artificial thread and process for making same.

L. LILIENFELD (U.S.P. 1,683,199, 4.9.28. Appl., 23.12.26. Austr., 20.6.25).—See B.P. 294,521; B., 1927, 745.

Artificial material and process for making same.

L. LILIENFELD (U.S.P. 1,683,200, 4.9.28. Appl., 3.6.26. Austr., 20.6.25).—See B.P. 281,351; B., 1928, 228.

Treating crêpe waste.

G. BONNARD (U.S.P. 1,683,520, 4.9.28. Appl., 30.4.27. Fr., 10.5.26).—See B.P. 270,727; B., 1927, 552.

Production of waterproof textile material, paper, etc. C. KNOPF (U.S.P. 1,683,470, 4.9.28. Appl., 24.4.25. Ger., 23.12.24).—See B.P. 251,126; B., 1926, 627.

Testing the quality of cotton fibres and the like. G. ZWEIFLE (B.P. 283,861, 16.1.28. Ger., 17.1.27).

Apparatus for measuring the moisture content in wool and like fibrous materials. ROYLES, LTD., C. WILSON, and E. H. TOWNEND (B.P. 296,157, 25.6.27).

Liquid purifiers (B.P. 296,178).—See I.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Dyeing of regenerated cellulose silk. BRIT. DYE STUFFS CORP., LTD., R. BRIGHTMAN, and P. CHORLEY (B.P. 296,011, 22.4.27).—Level dyeings on viscose

silks are obtained by using dyes obtained by coupling tetrazotised 4:4'-diaminodiphenyl mono-, di-, or trisulphide with 2 mols. of an aminonaphtholsulphonic acid, or with 1 mol. of such acid and 1 mol. of any other coupling component. Examples are: 4:4'-diaminodiphenyl sulphide coupled with salicylic acid and phenyl- γ -acid (brown), with 2 mols. of phenyl-J-acid (bluish-red), or with phenylmethylpyrazolone and J-acid (scarlet); the disulphide with 2 mols. of J-acid (scarlet); the trisulphide with salicylic acid and *m*-xylyl- γ -acid (brown), or with 2 mols. of γ -acid coupled acid (maroon).
C. HOLLINS.

Production of yellow dyeings on cellulose esters or ethers. I. G. FARRENIND. A.-G. (B.P. 275,230, 25.7.27. Ger., 29.7.26).—Yellow non-phototropic shades on acetate silk are obtained by using arylazodiphenylamines containing one sulphonic group and in the diphenylamine residue at least one nitro-group, *e.g.*, 4-benzeneazo-2' : 6'-dinitrodiphenylamine-4'-sulphonic acid and 4-benzeneazo-4'-nitrodiphenylamine-2'-sulphonic acid.
C. HOLLINS.

Coloration of cellulose esters and ethers. Soc. CHIM. DES USINES DU RHÔNE, and M. J. THEUMANN (B.P. 294,137, 12.4.27. Addn. to B.P. 275,553; B., 1928, 639).—The prior process is extended to include the formation of lakes of organic dyes in solutions of cellulose esters or ethers in an organic solvent, the coloured product being subsequently precipitated with water and washed. *E.g.*, a slight excess of an aqueous solution of lead acetate may be stirred into an acetone solution of cellulose acetate coloured with eosin, and the resulting product precipitated etc. as described.
D. J. NORMAN.

Dyeing throughout with vat dyes compact vegetable material. Soc. CHEM. IND. IN BASLE (B.P. 288,306, 4.4.28. Switz., 9.4.27).—The material (*e.g.*, twisted, highly mercerised embroidery and knitting yarn) is treated with a protective colloid such as glue or sulphite-cellulose lye in hot water before dyeing. Good penetration and even dyeings may then be obtained with vat dyes of the anthraquinone series, *e.g.*, Cibacine Blue 3G or Indanthrene Blue Green B.
C. HOLLINS.

Reducing deterioration of colours of textile fabrics during washing. HENKEL & Co., G.M.B.H. (B.P. 276,337, 16.8.27. Ger., 17.8.26).—If carbamide is added to detergent liquors for textiles, running of colours is largely prevented, the colours are brightened, and the "new" touch of silks and wool is restored.
C. HOLLINS.

Fabric dyeing processes. J. C. WATSON (B.P. 295,982 and 295,977, 18.5.27).—See U.S.P. 1,629,769 and 1,629,770; B., 1927, 553.

Mounting textile bobbins to be dyed in columns. J. ANNICQ (B.P. 296,603, 5.12.27).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Explosive properties of solid hypochlorites. J. WEICHERTZ (Chem.-Ztg., 1928, 52, 729—730).—Pure calcium hypochlorite with 70% of available chlorine evolves oxygen slowly at temperatures below

100°, but explodes violently with liberation of its oxygen content at 112°. Mixtures of the hypochlorite with organic substances such as alcohol, tars, mineral oils, or starch are stable at ordinary temperatures, but, on warming, deflagrate, sometimes with explosive violence. Similar effects are obtained by contact with moisture, but in this case there is a more or less lengthy period of induction depending on the nature of the organic substance.
A. R. POWELL.

Manufacture of zinc oxide. R. G. DANIELS (J. Oil Col. Chem. Assoc., 1928, 11, 277—288).—Oxidation of zinc in crucibles each with its own furnace gives good quality oxide if very pure zinc is used, but the thermal efficiency is low. Cadmium and lead give rise to discoloration. Cadmium can be removed by preheating in a reducing atmosphere, but fuel costs are prohibitive. A converter similar to the Bessemer converter consisting of a steel shell with chrome brick lining and hand turning gear was constructed and gave good results. Zinc being fed in the molten state, the heat of oxidation was more than sufficient and air cooling was necessary. For the manufacture of zinc oxide from residues and ores the Wetherill grate was used. This consists of a cast-iron plate with conical perforations tapering upwards placed over a brick chamber. Zinc ore and anthracite culm are fed on to the grate, which is worked by forced draught from below. Zinc sulphate solution (*d* 1.2) was used to bind the charge, which was briquetted, and recoveries of over 80% were obtained with residues and calamine. Zinc oxide discoloured with lead and cadmium is rendered white by heating with zinc sulphate at 600—720°. A deep charge is necessary. The impurities are converted into white basic sulphates. About six times the theoretical amount of zinc sulphate is required. Small proportions of lead in zinc oxide can be determined by dissolving in hydrochloric acid, adding ammonia solution, and saturating with hydrogen sulphide. The colour of the turbid solution is matched with that given by a pure zinc with known addition of lead.
C. IRWIN.

Corrosion of lead. JONES.—See X. **Potassium xanthate as fumigant.** DE ONG and TYLER.—See XVI.

PATENTS.

Manufacture of sulphuric acid in towers, chambers, and boxes. S. BARTH (G.P. 446,398, 11.7.23).—The Glover tower and a small part of the usual lead chamber are retained, whilst the greater part of the lead chamber is replaced by a series of absorption boxes through which the gases from the chamber are passed. These boxes are divided longitudinally into three long narrow compartments by means of perforated walls, and the gases are passed into the central compartment and out of the side compartments.
A. R. POWELL.

Contact sulphuric acid process. A. O. JAEGER, Assr. to SELDEN Co. (U.S.P. 1,675,308—9, 26.6.28. Appl., [A] 3.8.27, [B] 24.8.27).—Contact masses are made from (A) non-siliceous base-exchange materials, (B) zeolite mixtures, which have been combined with a catalytically active material such as vanadic acid. The absence of platinum enables the contact masses to be used in the presence of catalyst poisons.
C. HOLLINS.

Manufacture of phosphoric acid and phosphates.

I. E. WEBER, H. E. ALCOCK, and B. LAPORTE, LTD. (B.P. 295,848, 22.9.27).—Phosphoric acid made from a mineral phosphate and sulphuric acid, or the mixture containing phosphoric acid, is heated at 150–200° for some hours to destroy organic matter before the mass is worked up into the final product. W. G. CAREY.

Heat-producing composition. C. E. LYON, ASSR. to HOOKER ELECTROCHEM. CO. (U.S.P. 1,679,432, 7.8.28. Appl., 21.12.25).—Local heating without flame may be produced by addition of water to a mixture of 90 pts. of powdered sodium hydroxide and 10 pts. of anhydrous sodium aluminium chloride; addition of sodium chloride to the mixture serves to moderate the effect.

A. R. POWELL.

Preparation of colloidal metal solutions. C. MARTINESCU (G.P. 446,864, 18.2.25).—A dilute solution of a soluble salt of the metal is treated with thin aluminium foil whereby a dilute colloidal solution is obtained. This is enriched by suspending in it a more or less insoluble salt of the same metal and repeating the treatment with aluminium.

A. R. POWELL.

Production of base-exchanging substances. A. ROSENHEIM (B.P. 276,967, 21.7.27. Ger., 1.9.26. Addn. to B.P. 266,313; B., 1928, 710).—Material consisting entirely or in part of substances necessary for base-exchanging are vitrified, or, if already vitreous, are revitrified, with the addition of substances (*e.g.*, alkali carbonates, water-glass, etc.) which simplify vitrification; the vitrified portion is separated from the unvitrified and is then treated at increased pressure with solutions of substances containing the necessary base-exchanging constituents.

W. G. CAREY.

Decolorising agents. BÜTTNER-WERKE A.-G. and F. KLEINMANN (B.P. 295,623, 14.5.27).—Fuller's earth and finely-divided carbon are intimately mixed in the presence of a good wetting liquid, *e.g.*, alcohol, acetone, in a kneading machine or by heat-treatment, *e.g.*, boiling, so that the carbon particles do not separate during the decolorising process. Suitable binding agents with decolorising powers, *e.g.*, silica gel, may be added before treatment.

W. G. CAREY.

Production of acid substances used in the production of carbon dioxide. E. W. GEERE (B.P. 295,842, 9.9.27. Addn. to B.P. 276,146; B., 1927, 815).—A suitable liquid acid is added in excess to acid or other salts, which are then treated with fats, waxes, or hydrogenated oils, and finally with untreated acid salts according to the final acid strength required.

W. G. CAREY.

VIII.—GLASS; CERAMICS.

Coloured glasses. W. M. HAMPTON (J.S.C.I., 1928, 47, 192–196 r).—A general description is given of the properties and manufacture of coloured glasses. The factors controlling manufacture and the difficulties encountered are indicated. A summary of the methods of control of the colour and the effects of various colouring oxides is included.

PATENTS.

Ceramic material. F. H. RIDDLE, ASSR. to CHAMPION PORCELAIN Co. (U.S.P. 1,682,250, 28.8.28. Appl., 3.1.21).—Alumina is mixed with clay and a flux, in such amount as to give aluminium silicate on firing.

F. G. CLARKE.

Refractory ware. Ceramic material. F. H. RIDDLE, ASSR. to CHAMPION PORCELAIN Co. (U.S.P. 1,682,249 and 1,682,251, 28.8.28. Appl., [A] 29.11.20, [B] 9.9.22).—See B.P. 259,757; B., 1926, 1014.

Annealing sheet glass manufactured intermittently by means of sheet glass forming machines. SOC. ANON. DES MANUF. DES GLACES ET PROD. CHIM. DE ST. GOBAIN, CHAUNY, & CIREY (B.P. 296,282, 12.4.28. Fr., 18.2.28).

IX.—BUILDING MATERIALS.**PATENTS.**

[Brick] kilns. A. L. GELDENS (B.P. 295,867, 2.11.27).—The firebridges of the combustion chambers in a Hoffman type kiln are constructed to distribute the flames uniformly among the products being fired. Briefly, the arrangement consists of a shelf projecting away from the fire and cored with channels which act as jets to send the flames a considerable distance into the work chamber.

B. M. VENABLES.

Manufacture of hydraulic cement. DR. A. WACKER GES. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 282,458, 20.12.27. Ger., 21.12.26).—Dry calcium hydroxide obtained by making acetylene from calcium carbide with a small proportion of water is mixed with clay and the mixture is burnt.

W. G. CAREY.

Preservation of wood. W. IWANOWSKI and J. TURSKI (B.P. 296,332, 28.4.27).—Wood is impregnated with di-, tri-, or poly-chloro-derivatives of phenol or phenoxides, or with the condensation products of these compounds with arsenic chloride, alone or in admixture with other preservatives.

L. A. COLES.

Fireproof and anti-rotting composition [for wood]. T. SHIGA (U.S.P. 1,674,802, 26.6.28. Appl., 1.11.26. Japan, 17.7.26).—Boric acid is added to ammoniacal magnesium sulphate solution, followed by acetic acid and sodium cresolate.

C. HOLLINS.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Gaseous cementation of iron and steel. IV. **Action of mixtures of carbon monoxide and ammonia on iron and steel, and its bearing on the process of cementation.** A. BRAMLEY and G. TURNER (Iron Steel Inst. Carnegie Schol. Mem., 1928, 17, 23–66; cf. B., 1927, 485, 844). V. **Determination of iron-iron nitride eutectoid.** **Action of ammonia on steels containing different concentrations of carbon.** A. BRAMLEY and F. W. HAYWOOD (*Ibid.*, 67–87).—IV. The cementation of Armeo iron by mixtures of ammonia and carbon monoxide has been studied at temperatures between 700° and 1050°. The carburising action of the mixtures is much greater than that

of carbon monoxide alone. This is regarded as due to the formation of cyanides, which were detected experimentally, and to the effect of the hydrogen, formed by decomposition of the ammonia, on the carbon monoxide-carbon dioxide equilibrium. The depth of penetration and distribution of carbon during cementation closely resembled those previously found (*loc. cit.*) for cementations under corresponding conditions with mixtures of carbon monoxide and pyridine. The distributions of nitrogen, however, resembled those previously found with mixtures of carbon monoxide and acetonitrile, the greater stability of cyanides at high temperatures accounting for the large amount of nitrogen introduced. Hence comparatively little cyanide can be formed when pyridine decomposes at high temperatures. Mixtures of carbon monoxide and hydrogen were also used, and had carburising properties greater than those of carbon monoxide alone, but less than those of the corresponding mixtures with ammonia. The addition to carbon monoxide of one fourth its volume of hydrogen greatly increases the carburisation, but further additions of hydrogen have less effect, and a stage is eventually reached at which carburisation is reduced. When cemented above 900° the specimens decrease in length, but below 900° the specimens expand on cementation. The microstructures are profoundly influenced by the presence of nitrogen, especially below 800°; above 800° marked segregation of the cementite takes place. Needles of iron nitride are only present in zones in which the concentration of nitrogen is below 0.22% and above 0.015%.

V. A cylindrical bar of Armco iron was nitrogenised by heating it in a mixture of nitrogen and ammonia. The nitrogen concentration at different depths was then determined by turning part of the specimen down in a lathe and analysing the thin concentric layers of metal thus obtained. By combining these values with measurements of photomicrographs of a section of the same specimen, the composition of the iron-iron nitride eutectoid was determined as 2.0% N. If it be assumed that the laws of Raoult and Van't Hoff can be applied to the lowering of transition points of iron by the compounds Fe_3C and Fe_2N , the temperature of the iron-iron nitride eutectoid is deduced as 608°. If steels be used in place of iron, the amount of nitride formed decreases as the initial concentration of carbon increases, and slight loss of carbon takes place. No evidence could be found for the formation of complex compounds of iron, carbon, and nitrogen. Nitrogenised steel, free from carbon, will by suitable heat-treatments give rise to all the structures of carbon steels, although with a somewhat finer texture.

In an appendix the above-mentioned analytical microscopic method was used to determine the eutectoid composition in pure carbon steels, the value found being 0.89% C. The solubility of carbon in α -ferrite is deduced as 0.04% C, but this value depends on determinations of pearlite, and it is possible that thin films of cementite may still be present in a steel containing 0.04% C.

W. HUME-ROTHERY.

Specific heat of iron below 400°. W. H. DEARDEN (Iron Steel Inst. Carnegie Schol. Mem., 1928, 17, 89—108).—The sp. heat of Swedish Lancashire iron

(0.04% C, 0.006% Si, 0.006% S, 0.069% P) has been determined over the range 16° to 400° by a modification of the vacuum calorimeter method of Schwes and Günther (cf. Nernst, "The New Heat Theorem," 1926). The sp. heat rises rapidly from 0.0852 at 17° to a maximum of 0.1942 at 108°, then falls to about 0.12 in the range 150° to 200°, and then rises again. These values are subject to a very slight correction for the energy taken up by the small heating disc, but this does not affect the relative values. The abnormal maximum sp. heat at 110—115° corresponds to other abnormalities in sonority, resistivity, mechanical properties, etc., but the nature of the change is obscure.

W. HUME-ROTHERY.

Magnetic and electrical properties of cast iron. J. H. PARTRIDGE (Iron Steel Inst. Carnegie Schol. Mem., 1928, 17, 157—190).—The effects of different metals on the magnetic and electrical properties of cast irons have been investigated. The form in which the carbon is present greatly influences the magnetic properties. Graphite does not affect the hysteresis loss, but prevents the attainment of very high magnetic induction, especially if present in the form of flakes; if present as nodular graphite a higher induction can be obtained. The effect of graphite is due to lack of magnetic continuity. A ground mass of ferrite gives low hysteresis loss and high permeability, whilst a pearlite matrix gives high hysteresis loss and low permeability. Silicon decreases the magnetic induction, remanence, coercive force, and hysteresis loss of cast iron, and, in amounts not exceeding 5%, also decreases the maximum permeability. The effect on hysteresis loss is particularly marked, and alloys with a very low hysteresis loss can be cast. On annealing, the magnetic effects are complex according to the structure produced, but silicon increases the electrical resistance in both cast and annealed conditions. Cobalt increases the induction, remanent magnetisation, and permeability of cast iron, but has little effect on the coercive force and hysteresis loss, which remain high. Annealing increases the permeability, and decreases the hysteresis loss, coercive force, and remanent magnetism. Cobalt decreases the electrical resistance in the cast state, but the results for annealed specimens are erratic. Nickel decreases the permeability to such an extent that cast irons containing 15% Ni are practically non-magnetic. Nickel, and also aluminium, increases the electrical resistance of cast iron. Blackheart malleable iron possesses a high induction and permeability, with low hysteresis loss and coercive force; this is because nearly all the carbon exists as small islands of graphite in a ferrite matrix. Whiteheart malleable iron has a lower permeability and induction than blackheart, but the permeability is still greater than that of a mild steel. It is concluded that by using a suitable composition cast iron could in many cases be used in place of steel in the electrical industry, whilst the high electrical resistance is an additional advantage where it is desired to reduce eddy currents.

W. HUME-ROTHERY.

Apparatus for determination of sulphur in iron and steel. M. H. STEINMETZ (Ind. Eng. Chem., 1928, 20, 983).—In order to prevent too large a quantity

of hydrochloric acid and steam from distilling over into the absorption flask when determining sulphur in steel by the evolution method, the dissolution flask is fitted with a glass condenser by means of a sulphur-free rubber stopper. The condenser is provided with a central sealed-in tube with thistle funnel for adding the acid to the steel.

A. R. POWELL.

Precipitation method for determination of vanadium, and its application to steel analysis. B. S. EVANS and S. G. CLARKE (Analyst, 1928, 53, 475—486).—To 5 g. of steel are added 80 c.c. of dilute sulphuric acid (1:7) and dissolution is effected, as far as possible, by heating, after which nitric acid is added to clear the solution, followed by 70 c.c. of citric acid (100 g. in 200 c.c.) and sufficient hot concentrated sodium carbonate solution to render neutral or only faintly alkaline. Any tungstic oxide will then be in solution, and if nickel be present this must be separated by means of dimethylglyoxime (1 g. in hot alcohol). After filtering, 25 g. of sodium sulphite crystals are added, followed by 40—45 g. of commercial potassium cyanide dissolved in 100 c.c. of water. After shaking, the liquid is warmed for 10 min., boiled for 5 min., and the solution tested to make sure the iron is all present as ferrocyanide. Dilute sulphuric acid (1:3) is added to bring to neutral point (litmus), and the solution is shaken, set aside for at least 2 hrs. for the vanadium to settle out, and filtered through a "free-running" pulp filter, the precipitate being washed with 2% ammonium nitrate solution. Filter pulp and precipitate are ashed, fused with 1 pt. of sodium phosphate and 5 pts. of fusion mixture, and the melt is extracted with hot water, filtered, washed with dilute sodium sulphate solution, and the filtrate acidified with sulphuric acid, and boiled with a few drops of a saturated solution of potassium permanganate, and then boiled again for 20 min. after addition of 20 c.c. of saturated sulphur dioxide solution. Finally the liquid is titrated with 0.1*N*-potassium permanganate at 80°. No metals except nickel interfere with this process and accuracy to the limit of the permanganate titration is obtained, i.e., to about 0.1% of vanadium on a 5 g. sample. In the case of small amounts of vanadium the hydrogen peroxide method is used with a colorimetric process, but it is necessary to modify the procedure slightly, particularly in the removal of the molybdenum, the bleaching of any colour due to titanium, and the precipitation of the vanadium by cupferron if chromium interferes with the colour.

D. G. HEWER.

Natural rusting tests with cupriferous steel. K. DAEVES (Stahl u. Eisen, 1928, 48, 1170—1171).—Atmospheric rusting tests over a period of 21 months with a steel containing 0.03% Cu and with one containing 0.23% Cu showed the marked superiority of the latter. Ungalvanised wires of the former lost 23% and of the latter 16% in weight; the loss in weight with galvanised wires was 12% and 7%, respectively. The zinc coating of the copper-steel remained unperforated by rust for a much longer period than that of the same steel without copper, and the tendency to pit and scale was much less with the copper-steel.

A. R. POWELL.

WITTNEBEN (Z. Metallk., 1928, 20, 316—322).—As is the case with soft iron and copper, the curve showing the minimum temperature of recrystallisation against the minimum deformation required to cause recrystallisation to take place is a hyperbola for α -brass. The minimum temperature below which no amount of work will cause recrystallisation in α -brass increases with an increase in the zinc content from 265° with 10% Zn to 330° with 32% Zn. The axes of reference of the hyperbolæ showing the grain-growth against the time of annealing coincide with the co-ordinate axes for 500°, 550°, and 700°, but for 850° the abscissæ axis is displaced towards higher temperatures. The grain size after recrystallisation is independent of the original grain size and depends only on the time, temperature, and degree of deformation. The rate of recrystallisation of brass containing 90% Cu changes with the temperature according to the exponential curve $V = 1.0134t^{-26.5}$. An increase in the zinc content also accelerates recrystallisation at temperatures above 350°.

A. R. POWELL.

Corrosion of chemical lead. D. W. JONES (J.S.C.I., 1928, 47, 161—167 T).—Antimony is regarded as a very injurious impurity, and copper is one of the few metals which can practically be introduced into lead with any hope of beneficial results. Chemical lead is examined by immersion in either plain sulphuric acid (*d* 1.72) or nitrous vitriol (2.5% N_2O_3) of *d* 1.72. The general behaviour and loss in weight are recorded. Lead sulphate has an important bearing on corrosion, and the method can give a value to this form of protection. Addition of 0.027% Cu to lead containing 0.008% Bi has the effect of correcting a loss. In plain sulphuric acid pure lead can show a "protective coating value" of over 100%. A second immersion of pure lead after cleaning and exposing a fresh surface had the effect of reducing corrosion losses, possibly due to a state of passivity, or a measure of passivity, having been brought about in the lead. When nitric or nitrous acid is involved, a great decline is shown in the figure which represents the "protective coating value"; the average for all leads in plain acid was 70, whereas in nitrous vitriol it was only 3. Of the four leads which lost most by corrosion, three contained copper, whilst the fourth, a pure lead, failed to pass the "flash" test. There was no evidence to prove that selective action had proceeded with respect to the dissolution of copper in any lead. Over longer periods the "protective coating value" of coppered lead in plain sulphuric acid can exceed 100%, and this type of lead resists plain sulphuric acid at atmospheric temperature rather better than pure lead. The latter over long periods of immersion resists corrosion in nitrous vitriol to a greater degree than a coppered lead. A high standard of purity in lead (99.99%) is essential—except for copper, which is permitted in some cases to the maximum extent of 0.05%. Casting previous to rolling sheet must be conducted so that no "cold setting" takes place, and there must be no excessive shrinkage in the cast block which would show cleavage of lead crystals at those places which solidify last of all. Too much reliance is sometimes placed on the self-annealing characteristic of lead, with the result that it may be subjected to excessive working.

Rupture has been brought about by severely straining cold-worked lead. Aspects of corrosion related to users of chemical lead are: (1) Pitting, which is attributed in some cases to a too liberal use of nitre. (2) The necessity of properly supporting lead wherever applied because under stress or strain with or without the aid of a corrosive agent intercrystalline failure is induced. A grave danger lies in the introduction of copper to mask a fault, and uniformity in the final product may be lacking.

Aluminium and its alloys in the chemical and allied industries. J. DORNAUF (Z. angew. Chem., 1928, 41, 993—1001).—Results of corrosion experiments with pure aluminium and with its alloys together with a consideration of their physical properties and micro-structure are recorded. The value of the alloy "silumin" is emphasised. A *précis* is presented of data concerning the corrosion of a large number of aluminium articles of industrial use.

A. G. POLLARD.

Corrosion of aluminium conductor wires. H. BOHNER (Z. Metallk., 1928, 20, 309—315).—As a result of an investigation into the causes of various corrosion phenomena which may occur in aluminium conductor wires, the conclusion is reached that failure may be ascribed to four main causes, viz., the presence of too much impurity or the segregation of impurities due to incorrect heat-treatment, careless work in the wire-shops involving the introduction of foreign metals into the wire or "lapping," incorrect methods of suspending the wires, and, finally, changes in the structure of the wires due to lightning or short-circuits. To avoid these troubles it is recommended to use 99.5% aluminium; before drawing, the forged castings should be annealed just below the solidus point to obtain complete solid solutions of the impurities and then quenched. During drawing, the wire must be annealed below 200°, and, after drawing, for 7—20 hrs. at 175°. A. R. POWELL.

Hardness and its relation to the cold-working and machining properties of metals. II. H. O'NEILL (Iron Steel Inst. Carnegie Schol. Mem., 1928, 17, 109—156; cf. B., 1927, 490).—The construction of a sclerometer suitable for use with both hard and soft metals is described. In this the scratch is made by a 1 mm. hemispherical diamond, the surface of the specimen being tilted at an angle of 7° so that the scratch is made downhill. The load is applied by means of weights. Tests made with this instrument on single crystals of iron and on polycrystalline iron and steels are described. The mean pressure hardness values obtained are of the same order as those given by the Brinell test, and the method is suitable for the study of directional hardness in single crystals. If the orientation of an unstrained body-centred or face-centred cubic crystal be determined by making pressure figures, the scratch hardness is greatest in the direction parallel to the lobes of the pressure figure. The effect of straining large crystals has been studied. The relations between the various hardness tests and the "machinability" of metals are discussed. No hardness test alone is able to place metals in the order of their machinability; the micro-structure and other factors have always to be considered.

But the Brinell value H_u (the Brinell number corresponding to the load which gives an indentation of the same diameter as the indenting ball) gives a much better indication of the machinability than the ordinary Brinell number. The relations between indentation, scratch, tensile, and compression tests are discussed. There is a similarity between the curves connecting true stress with some function of strain irrespective of the method by which deformation is caused. The phenomena are correlated under the title "pressure of fluidity" introduced by Ackerman (Trans. Soc. Engineers, 1923, 25). The historical development of the term "hardness" and hardness testing is summarised; both elastic and plastic deformation should be measured, and an instrument for this purpose is described.

W. HUME-ROTHERY.

Ludwig-Soret effect in metallic alloys. M. BALLAY (Rev. Mét., 1928, 25, 427—454).—The Ludwig-Soret effect has been measured in binary liquid alloys of tin with lead, copper, cadmium, and zinc by heating in vertical and horizontal silica tubes, the temperature difference being of the order of 500°. The effect is the greater the larger is the temperature gradient. About 150 hrs.' heating was required to reach equilibrium. In horizontal tubes no differences in composition are produced unless precautions are taken to avoid convection, by making a constriction at the centre of the tube. The effects then observed are small, and may be in the reverse sense to those obtained in vertical tubes. Solid alloys of lead with tin and with thallium, when treated similarly, showed only very small effects. A wire of platinum-rhodium alloy showed a distinct effect after heating over a Méker burner for 55 days.

C. W. GIBBY.

Determination of copper in molybdenite. M. G. RÆDER (Tidssk. Kjemi Bergvesen, 1928, 8, 91—93).—1 g. of molybdenite concentrates (which should not contain less than 0.02 g. Cu) is dissolved at a gentle heat in 25 c.c. of nitric acid (3:1) and 6—7 c.c. of concentrated sulphuric acid, and the liquid evaporated to strong fuming. The residue is boiled with 100 c.c. of water, and, after filtering, the filtrate is diluted to 200—300 c.c. and heated to boiling. *N*-Sodium thio-sulphate is added from a burette until the deep blue solution shows a turbidity due to separated sulphur. After again boiling, the liquid is filtered and the precipitate washed with water containing sulphur dioxide until the filtrate is colourless. The paper and precipitate are ignited, and the copper in the residue is determined by titration with 0.02*N*-sodium thiosulphate. The above method yields accurate results, and is quicker than the usual separation with thiocyanate. H. F. HARWOOD.

Trend of engineering developments in steel. B. D. SAKLATWALLA (J.S.C.I., 1928, 47, 198—202, 210—212 t).

Production and uses of beryllium. K. ILLIG (Trans. Amer. Electrochem. Soc., 1928, 54, Advance copy, 211—222).

Preservation of metals. BORODULIN.—See II. **Corrosion of copper by milk.** QUAM and others.—See XIX.

PATENTS.

Cupola with forehearth. H. LUYKEN (U.S.P. 1,681,043, 14.8.28. Appl., 26.2.24. Ger., 5.3.23).—The lower end of the cupola is connected with the upper part of the forehearth through a vertical channel with two horizontal channels of small cross-section on opposite sides thereof, so as to damp momentary fluctuations of the level of the metal in the shaft.

A. R. POWELL.

Open-hearth furnace. A. T. KELLER, ASSR. to BETHLEHEM STEEL CO. (U.S.P. 1,682,378, 28.8.28. Appl., 5.12.21).—An end construction for open-hearth steel furnaces comprises a series of air-gas channels with horizontal air and gas ports terminating near the end of the combustion chamber, a separate port for waste gases, and means for closing the terminal ends of the air and gas ports to protect them from the hot flue gases.

A. R. POWELL.

Regenerative open-hearth furnaces. S. J. CORT, T. BURNS, R. S. A. DOUGHERTY, and C. E. LEHR (U.S.P. 1,680,365, 14.8.28. Appl., 24.9.25).—A system of buckstaves and beams provided with water-cooling is described.

B. M. VENABLES.

Smelting furnace. J. A. LANIGAN (U.S.P. 1,682,343, 28.8.28. Appl., 15.8.27).—The furnace comprises a cupola shaft with fuel door above the tuyères and a sloping bottom communicating with the hearths of a reverberatory furnace, which is heated from the usual firebridge and from inclined tuyères in the roof, one of which is directed over the communicating channel between the shaft and hearths.

A. R. POWELL.

Apparatus [furnace] for smelting. H. W. HALL, ASSR. to AMER. SMELTING & REFINING CO. (U.S.P. 1,681,535, 21.8.28. Appl., 30.7.25).—A furnace of the reverberatory type is provided with feed openings on each side in which rotating cylinders provided with shovelling devices are operated in such a manner that the charge is continuously fed to the furnace at the top and forms an inclined heap in the furnace, which closes the charging opening.

A. R. POWELL.

Rotary drum furnace for roasting sulphur-containing ores and the like. METALLBANK & METALLURGISCHE GES. A.-G. (B.P. 284,731, 4.2.28. Ger., 4.2.27).—A rotary drum furnace is provided with a number of vertical partitions, with central apertures, dividing the furnace into a number of compartments. Lifters are provided on the cylindrical wall, also projections, guide members, etc. to break up the material as it is showered. Transfer of the material through the central apertures is effected by inclined guide members supported on a fixed hollow shaft projecting through the end closures of the furnace. The hollow shaft serves also to introduce the air for roasting; the inclined guides are adjustable to vary the rate of transfer of the material. The partition walls may be secured either to the rotating part of the furnace or to the fixed hollow shaft.

B. M. VENABLES.

Furnace for treating metal. W. E. WATKINS, ASSR. to COPPER PLATE, SHEET & TUBE CO. (U.S.P. 1,679,389, 7.8.28. Appl., 25.6.27).—The furnace consists of a long horizontal chamber with a horizontal dividing wall at the front end to divide this part into a

lower combustion chamber and an upper preheating zone.

A. R. POWELL.

Apparatus for reclaiming [easily oxidisable] metals [from dross]. T. D. STAY and C. O. TESSIER, ASSRS. to ALUMINUM CO. OF AMERICA (U.S.P. 1,679,385, 7.8.28. Appl., 20.10.25. Cf. U.S.P. 1,630,361; B., 1927, 528).—The dross is heated just above the m.p. of the metal in a crucible provided with a rotating propeller so shaped as to cause the dross to rise to the surface while the liberated metal is thrown against the walls of the crucible. The dross is discharged on to a vibrating screen from which it passes through a launder in which the liberated metal is agglomerated and rapidly solidified. Finally, a second vibrating screen separates the coarser particles of metal from the finely-divided oxide dust.

A. R. POWELL.

Liquid treatment [quenching] of roasted ores. M. E. BRANTHAVER, ASSR. to AMER. MILLING & REFINING CO. (U.S.P. 1,681,696, 21.8.28. Appl., 10.5.24).—The roasting furnace is provided with a water-sealed discharging duct which delivers the hot ore to the quenching pit without permitting access of air.

A. R. POWELL.

Flotation process. D., M., S. R., and S. GUGGENHEIM, J. K. MACGOWAN, and E. A. C. SMITH (GUGGENHEIM BROS.), ASSEES. of A. H. FISCHER (B.P. 296,270, 5.3.28. U.S., 29.12.27).—Ethyl xanthylformate, $\text{EtO} \cdot \text{CS} \cdot \text{S} \cdot \text{CO}_2\text{Et}$, m.p. -36.5° , obtained from ethyl chloroformate and potassium xanthate, is used as collecting agent in conjunction with a frothing agent (cresylic acid, pine oil, etc.) in ore flotation processes. [Stat. ref.]

C. HOLLINS.

Manufacture of wrought iron. H. WADE. FROM INTERNAT. NICKEL CO. (B.P. 295,857, 11.10.27).—An alloy of iron with 0.25–5% Ni, 0.08% C, and 0–1% Cu, Cr, or Mo is claimed. [Stat. ref.]

A. R. POWELL.

Alkali or alkaline-earth fluxes for deoxidation of iron and steel. MANNESMANNRÖHREN-WERKE (G.P. 448,510, 29.11.24).—The use of one or more alkali or alkaline-earth salts of boric, tungstic, or titanous acids, together with a flux, such as soda-lime glass or analcime, and a deoxidiser is claimed.

A. R. POWELL.

Production of a rust-preventing layer on iron and steel. W. SCHMIDTING (G.P. 448,009, 1.6.26).—The surface of the metal is treated with a solution of phosphoric acid, hydrogen peroxide, and potassium chlorate.

A. R. POWELL.

Manufacture of metal-coated sheets and plates. J. McFETRIDGE, ASSR. to AMER. SHEET & TIN PLATE CO. (U.S.P. 1,679,435, 7.8.28. Appl., 17.1.27).—Sheets of iron or steel for galvanising or tinning are transferred directly from the hot acid pickling bath to a bath containing a cold solution of suitable inorganic salts which act as fluxes in the subsequent galvanising or tinning operation.

A. R. POWELL.

Plating of metal. [Coating iron articles with copper.] W. E. WATKINS (B.P. 282,624, 5.7.27. U.S., 24.12.26).—The articles are coated with a suspension of finely-divided copper or copper oxide and carbon in a hydrocarbon oil with asphaltic base, then

heated in a tunnel kiln with a neutral atmosphere so that the products of combustion encircle the articles. The articles are thereby coated with a bright deposit of copper below a layer of iron oxide which is subsequently removed by pickling. [Stat. ref.] A. R. POWELL.

Additional material for ferrous metals. P. D. MERICA and T. H. WICKENDEN, ASSRS. to INTERNAT. NICKEL CO. (U.S.P. 1,680,058 and 1,680,161, 7.8.28. Appl., [A] 28.4.25, [B] 10.3.24).—(A) The properties of cast iron and other ferrous alloys are improved by the addition of chromium, manganese, tungsten, copper, or molybdenum, together with an alloy of nickel with sufficient carbon and silicon to reduce its m.p. to that of the remainder of the alloy. (B) About 5% of nickel containing 0.25–2.5% C and 1–10% Si is added to molten cast iron just prior to casting.

A. R. POWELL.

Treatment of alloy steel. A. C. DAVIDSON, ASSR. to D. Co., INC. (U.S.P. 1,680,937, 14.8.28. Appl., 28.10.19).—A mixture of iron, chromium, and tungsten in the desired proportions is melted, deoxidised under a layer of fluxes, treated with cobalt, again deoxidised, treated with vanadium in quantity not exceeding a 50% excess over the amount desired in the final alloy, and cast above 1750°.

A. R. POWELL.

Recovering copper from its ores. A. ELLIOTT (U.S.P. 1,681,528, 21.8.28. Appl., 17.3.24. Renewed 23.6.27).—Copper ores are leached with ferrous sulphate solution in a vessel through which a finely-divided stream of compressed air is passed.

A. R. POWELL.

[Nickel-copper] alloys. CANAD. GEN. ELECTRIC CO., LTD., ASSEES. of E. T. ASP (Can. P. 266,292, 2.3.25).—Copper-nickel alloys containing at least 50% Ni, 0.5–5% Al, and 0.1–1% Mg are claimed. The alloys are non-magnetic, work easily, and have a higher electrical resistance than similar alloys without the aluminium.

A. R. POWELL.

Treating copper alloys [brass and Muntz metal]. V. O. HOMERBERG and D. N. SHAW (U.S.P. 1,680,045—6, 7.8.28. Appl., [A] 28.1.26, [B] 30.1.24).—(A) Copper alloys, the composition of which falls within that portion of the temperature-composition diagram of the series which resembles the portion of the copper-zinc diagram in which 60:40 brass is found, are heated to such a temperature that a single solid solution is formed, quenched, aged at a temperature at which a constituent separates from this solution in a finely-divided form, and cooled at a rate which maintains this structure at the ordinary temperature. (B) Muntz metal is heat-treated as described above and aged at 250° to obtain a tensile strength of over 75,000 lb./in.² and a Brinell hardness of over 200.

A. R. POWELL.

Vertical zinc retort. F. G. BREYER and E. H. BUNCE, ASSRS. to NEW JERSEY ZINC CO. (U.S.P. 1,680,726, 14.8.28. Appl., 5.3.27).—An externally heated vertical reducing chamber for the distillation of zinc from its oxide is supported and tied in at its lower end only, and built up from refractory bricks to make a vapour-tight chamber.

A. R. POWELL.

Recovery of zinc from fine zinc ores etc. METALLBANK U. METALLURGISCHE GES. A.-G. (G.P. 448,598, 7.2.26.

Addn. to G.P. 421,384; B., 1926, 412).—The desulphurised and sintered ore is smelted together with the necessary carbonaceous material in a zinc furnace with vertical retorts.

A. R. POWELL.

Treatment of lead-zinc ores containing iron and manganese. E. LANGGUTH (G.P. 448,150, 7.7.25).—The ore is introduced into a fused mass of zinc chloride and alkali chloride, and when the reaction is completed the greater part of the lead is precipitated by addition of zinc. The salt mixture is then dissolved in water and the solution after addition of zinc oxide is electrolysed, whereby the iron and manganese are precipitated as oxides on the anode and the remaining lead together with some zinc is deposited as a sponge on the cathode. This sponge is utilised for the removal of lead from further quantities of the fused salts.

A. R. POWELL.

Fuel-heated furnace and converter [for nickel matte] using preheated air. O. LELLEP, ASSR. to INTERNAT. NICKEL CO. (U.S.P. 1,680,155, 7.8.28. Appl., 29.10.23).—A converter comprises a rectangular furnace for the molten matte above which is a cylindrical feeding chamber charged with roughly crushed matte and fluxes. A blast of air is blown into the lower chamber, where it is preheated, and passes upwards through the feeding chamber, thereby melting its charge, which falls through to the furnace chamber for further treatment.

A. R. POWELL.

Metallurgy of ores or materials containing tin and extraction of metals or metal products therefrom. E. A. ASHCROFT (B.P. 295,805, 7.7.27).—The ore or concentrate is heated at 700–900° with nickel pellets or nickel oxide and a reducing agent, such as carbon or sponge iron, together with a small quantity of zinc, iron, or ammonium chloride to act as catalyst or carrier. The tin is reduced to metal, which is carried in the form of chloride to the iron and nickel particles, with which it alloys, and the resulting iron-nickel-tin alloy is separated by means of an electromagnet. The tin is recovered from the alloy by electrolysis or by chemical means.

A. R. POWELL.

Aluminium alloys. R. SELZ (B.P. 266,720, 22.2.27. Ger., 27.2.26).—An alloy of aluminium with at least 18% of zinc is treated with 2% of a hardener made by melting 90 pts. of copper with 5 pts. of nickel under a flux of potassium fluoride and adding 5 pts. of magnesium.

A. R. POWELL.

Bearing-metal alloys. T. GOLDSCHMIDT A.-G. (B.P. 266,696, 9.2.27. Ger., 25.2.26).—The alloys comprise eutectic mixtures of aluminium with one or more of the following: silicon, copper, nickel, cerium, calcium, barium, strontium, or magnesium silicide, hardened with small quantities of lithium, magnesium silicide, or lithium silicide. Also there may be added titanium, vanadium, molybdenum, tungsten, chromium, iron, manganese, or boron. [Stat. ref.]

A. R. POWELL.

Improving the mechanical properties of heavy metals [nickel, cobalt, and lead] or their alloys. L. MELLERSH-JACKSON. FROM SIEMENS & HALSKE A.-G. (B.P. 295,971, 21.5.27).—The tensile strength and hardness of nickel, cobalt, lead, or nickel-copper alloys with more than 10% Ni are considerably increased by

the addition of 0.1–15% of beryllium together with small quantities of magnesium, tin, zinc, antimony, aluminium, cadmium, carbon, or silicon. The worked alloys are annealed at 600–900°, quenched, and aged at 150–600°. A suitable nickel alloy contains 92.5% Ni, 2% Be, 5% Si, and 0.5% C. Addition of 0.2% Be increases the strength of lead by 100% without heat-treatment.

A. R. POWELL.

Manufacture of molybdenum alloying compounds. E. A. LUCAS, Assr. to MOLYBDENUM CORP. OF AMERICA (U.S.P. 1,681,123—4, 14.8.28. Appl., 30.12.24).—(A) A composition for use in preparing molybdenum alloys comprises a fused mass of molybdenum trioxide and lower oxides with a basic oxide as a flux. (B) Molybdenite is roasted to expel sulphur and the product is fused with a reducing agent, a flux, and lime to form a coherent fused mass containing a calcium molybdenum silicate.

A. R. POWELL.

Coloration of metal surfaces. K. BARANOWSKI (B.P. 295,889, 21.12.27).—The articles are pickled, subjected to the action of a suitable current in an electrolytic bath to give a granular metal surface, sprinkled with colouring solutions or pastes, and heated in a more or less oxidising flame.

A. R. POWELL.

Manufacture of metal sheets and articles with radioactive properties. C. BALLIN (G.P. 448,160, 16.8.25).—Radioactive oxides are melted at a high temperature with iron, aluminium, copper, brass, or tin, and the products are rolled into sheets; e.g., 40 g. of uranium oxide are melted with 60 g. of tin, and the cast product is rolled.

A. R. POWELL.

Radioactive material. A. FISCHER (B.P. 285,467, 22.9.27. Austr., 19.2.27. Addn. to B.P. 278,347; B., 1928, 489).—Radioactive substances, e.g., radium salts, are introduced into short narrow platinum tubes, sealed at one end, the tube is then fused, welded, or soldered to close it above the substance introduced, and finally the container so produced is heated to bright redness whereby the radioactive metal enters into solid solution in the platinum.

A. R. POWELL.

Etching process [for metals] with electrolytically produced protective films. J. RIEDER (G.P. 448,554, 9.10.25).—The design is produced on the plate in the usual way, and the parts which are not to be etched are provided with a protective film of lead dioxide or manganese dioxide by anodic electrolysis. The design is then etched and the protective film removed by cathodic electrolysis in a strongly alkaline electrolyte.

A. R. POWELL.

Magnetic [chromium-iron] alloys. W. S. SMITH, H. T. GARNETT, and J. A. HOLDEN (B.P. 296,137, 30.4.27).—Alloys of high permeability for loading telephone conductors consist of iron with 1–10% Cr, 1–4% Al, and 0–1% Mn and/or Si. Part or all of the chromium may be replaced by molybdenum. High permeability is obtained by annealing at 900–1020° in an inert atmosphere and cooling slowly.

A. R. POWELL.

Production of metals in electric furnaces. E. G. T. GUSTAFSSON (B.P. 278,005, 26.9.27. Swed., 27.9.26).—During the main part of the operation of smelting a metalliferous charge, the current supply to the furnace

is adjusted to correspond to a combined arc and slag resistance not less than 0.020 ohm per electrode.

J. S. G. THOMAS.

[Roller] method of plating iron and other metals in the form of sheets or strips. F. JORDAN (B.P. 296,474, 1.6.27).

Magnetic core (G.P. 447,243).—See XI.

XI.—ELECTROTECHNICS.

Electrification in dust clouds. BLACKTIN.—See II.
Properties of cast iron. PARTRIDGE. **Corrosion of aluminium wires.** BOHNER.—See X.

PATENTS.

Electric induction furnaces. E. F. NORTHRUP (B.P. 296,464, 30.5.27).—In plant comprising a number of induction furnaces in parallel, individual power-factor means connected with the respective inductor coils and means for connecting power supply to the inductor coils of the various furnaces over a different set of turns from these rectifying devices are provided.

J. S. G. THOMAS.

Induction furnace. R. B. LINCOLN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,682,388, 28.8.28. Appl., 21.3.27).—The furnace comprises a helically-wound, water-cooled coil in which a crucible is nested, the whole being embedded in refractory material in a metal casing within which is a ferromagnetic device to prevent induction of heating currents in the casing.

A. R. POWELL.

Electric battery cell. II. KÖHNE and W. SUHRCKE (B.P. 296,184, 28.7.27).—In a cell of the Bunsen type, the positive electrode consists of a carbon rod supported within a glass, casein, or porcelain cylinder closed below by a carbon bottom and containing acid. The negative electrode of zinc or zinc-lead alloy is coated with a perforated sleeve.

J. S. G. THOMAS.

[Photo-sensitive surface for] photo-electric cells and other electric discharge devices. WESTINGHOUSE ELECTRIC & MANUF. CO., Assees. of V. K. ZWORYKIN (B.P. 291,763, 2.6.28. U.S., 8.6.27).—The active surface comprises superposed layers of different materials, the whole being transparent to light. Thus a thin film of alkali metal may be deposited upon a film of a more stable metal, e.g., tungsten or molybdenum.

J. S. G. THOMAS.

Making of electric conductors. J. V. CAPICOTTO, Assr. to DUBILIER CONDENSER CORP. (U.S.P. 1,683,064, 4.9.28. Appl., 27.2.24).—The conductor comprises an intimate mixture of finely-divided platinum and iridium containing also barium and strontium.

Welding electrodes. O. Y. IMRAY. From E.M.F. ELECTRIC CO. PROPRIETARY, LTD. (B.P. 296,509, 28.6.27).—A metallic core is coated with a hydrated silicate, e.g., china clay, and a carbonaceous material, e.g., vegetable black, so that hydrogen is produced by decomposition of water liberated from the hydrated silicate when the electrode is in use, and protects the fused metal of the weld against oxidation. [Stat. ref.]

J. S. G. THOMAS.

Manufacture of a magnetic core material. DEUTSCH-LUXEMBURGISCHE BERGWERKS- U. HÜTTEN-A.-G.,

and A. SCHNEIDER (G.P. 447,243, 8.6.23).—Hammer or rolling scale is pulverised and reduced to metal in a current of hydrogen. The iron powder so obtained is mixed with an insulating substance and the mixture pressed into cores. A. R. POWELL.

Electric arc lamp. I. J. LAVOISIER, Assr. to BURDICK CORP. (U.S.P. 1,682,847, 4.9.28. Appl., 1.10.24).—The lamp contains an arc electrode comprising a dehydrated and glazed mixture of zirconia and yttria.

F. G. CROSSE.

Chromium plating bath. ELEKTRO-CHROM-GES. M.B.H. (G.P. 448,526, 22.7.24).—A solution of chromium trioxide containing other acids in amount not exceeding 1.2% of the weight of chromium trioxide present is reduced cathodically in such a way that no metal is deposited. A. R. POWELL.

Pyro-electrical separation of finely-divided materials. G. A. OVERSTROM (U.S.P. 1,679,739 and 1,679,740, 7.8.28. Appl., [A] 24.11.25, [B] 23.11.26).—(A) Electrical separation of powdered materials is effected without external electricity, the materials being heated so that one constituent becomes more pyro-electric than another; the constituents separate as they pass over and fall off a surface. (B) A mixture of sand and mica is heated as above and allowed to slip over a glass surface. B. M. VENABLES.

[Control of temperature in] electric annealing resistance furnace. A.-G. BROWN, BOVERI & Co., (B.P. 278,352, 23.9.27. Ger., 30.9.26).

Gas-filled electric incandescence lamps [with concentrated filaments]. N. V. PHILIPS' GLOEILAMP-ENFABR. (B.P. 287,563, 19.3.28. Holl., 25.3.27).

Water-cooled mercury vapour arc lamps. A. K. CROAD. From HANOVIA CHEM. & MANUF. Co. (B.P. 296,527, 27.7.27).

Chromium plating [of recessed articles]. J. C. PATTEN (B.P. 282,337, 27.5.27. U.S., 20.10.26).

Temperature of gases (B.P. 274,440). **Purification of gases** (G.P. 446,862).—See I. **Treatment of lead-zinc ores** (G.P. 448,150). **Colouring metal surfaces** (B.P. 295,889). **Radioactive metal sheets etc.** (G.P. 448,160). **Etching process** (G.P. 448,554). **Magnetic alloys** (B.P. 296,137). **Production of metals** (B.P. 278,005).—See X. **Irradiation of liquids** (B.P. 279,902).—See XIX.

XII.—FATS; OILS; WAXES.

Component glycerides of coconut and palm-kernel fats. G. COLLIN and T. P. HILDITCH (J.S.C.I., 1928, 47, 261—269 T).—Oxidation of these fats by potassium permanganate in acetone solution according to the method of Hilditch and Lea (A., 1928, 152), together with fractionation analyses of the methyl esters of the fatty acids in the original fat and in the neutral product of oxidation (fully-saturated glycerides), leads to information of a partially quantitative nature as to the structure of the glyceride mixtures present. Coconut fat was found to contain 84%, and palm-kernel fat 63%, of fully-saturated glycerides. From the saponification equivalents of the latter and of the

original fats, and knowing the original content of unsaturated acids (8 and 18%, respectively, in coconut and palm-kernel fatty acids), it was found that in each, in the mixed saturated-unsaturated glycerides, one equivalent of unsaturated acid was present along with 1.3—1.4 equiv. of saturated fatty acids; this figure was confirmed by the results of the detailed methyl ester analyses. The proportions of the individual saturated acids in the mixed saturated-unsaturated glycerides have at present to be estimated by comparison of the amounts present in the original fat and in the fully-saturated glycerides. Since the amounts of several of the acids are relatively small, the small differences then obtained tend to make the data in question less accurate than when substantial proportions are present. The definite results obtained are as follows:—(i) Throughout both fats the fatty acids are distributed in the glycerides in a remarkably even manner, and no evidence has been obtained of the presence of any simple triglyceride, such as trilaurin or triolein. (ii) The unsaturated acids, as stated, are linked with 1.3—1.4 equiv. of saturated acids in both fats; since no triolein is apparently present, this corresponds with a mixture of about 3 equiv. of mono-oleo-disaturated glycerides with 1 equiv. of dioleomonosaturated glycerides. (iii) Subject to a few minor exceptions, the composition of the saturated fatty acids is of much the same order in both the fully-saturated and mixed saturated-unsaturated portions of the fats. (iv) Fractional crystallisation of the fully-saturated glycerides did not yield any one individual component, but the partial separation effected indicated that the most sparingly soluble components (judged from their equivalents and m.p.) were of the nature of dilauro-myristin, laurodimyristin, dilauropalmitin, lauromyristopalmitin, etc. (v) Practically the only difference between the two fats (apart, of course, from their differing content of oleic acid and the equally characteristic contents of their various saturated acids) was that, whilst but little palmitic acid was found in the mixed saturated-unsaturated glycerides of coconut fat, this acid was present in relatively large amount in the corresponding glycerides of palm-kernel fat. Less certain differences were observed in the distribution of caprylic and capric acids in these fractions, capric acid appearing to be absent from the unsaturated glycerides of coconut oil. The results of the investigation were compared with those obtained by Bömer (cf. B., 1924, 478) who adopted an entirely different procedure, namely, intensive fractional crystallisation of the two fats, and the conclusions arrived at by both methods appear on the whole to be compatible.

Tests for incipient rancidity of fats. W. L. DAVIES (J.S.C.I., 1928, 47, 185—187 T).—A survey of the more important tests for following the course of autocatalytic oxidation of fats is given and the drawbacks of the Kreis test are dealt with. A test to forecast the keeping qualities of batches of fats is described. Equal weights of fat are emulsified in 10 c.c. of diluted separated milk containing 1 c.c. of 0.25% methylene-blue, and the blue colour is bleached by the "reductase" of the milk by incubating at 37—40°. When bleached, the tubes are shaken for 15 sec. and kept for 2 min. before the intensity of the blue colour restored is noted. The

restoration of the blue colour compares with the oxidation intensity in the fats. The test was carried out on a large number of fats of known history and keeping qualities, and the restoration of colour corresponded accurately with the keeping qualities of the fats. The test was particularly applicable to detecting metallic contamination and comparing the catalytic activity of metals in oxidising fats. The test was also applied to study the state of oxidation of individual fats, and was found more reliable and less cumbersome than some of the other tests tried.

Luminescence of oils and fats. A. VAN RAALTE (Chem. Weekblad, 1928, 25, 544—546).—The appearance after refining of luminescence in fats and oils which show no luminescence in the crude condition is shown to be due to the removal by the refining process of some constituent which inhibits luminescence. It is found that this "anti-luminescent" constituent is removed by treatment of the oil or fat with fuller's earth, or with warm benzoyl peroxide, but not with cold benzoyl peroxide or norite. It is suggested that this constituent is a vitamin, and experiments in support of this view are described. S. I. LEVY.

Rapid determination of fatty acids in soap. N. SPASSKI (Masloboino-Shirowoje Djelo, 1926, [12], 33—34; Chem. Zentr., 1927, II, 1908).—An aqueous solution of the soap is acidified with hydrochloric or sulphuric acid, and the liberated fatty acids are extracted with ether. The ethereal solution is evaporated and the residual acids are dissolved in alcohol and titrated with 0.5*N*-alcoholic potash. The neutral fat is saponified with 0.5*N*-potash and the excess alkali titrated with 0.5*N*-hydrochloric acid, phenolphthalein being used as indicator in both titrations.

A. R. POWELL.

PATENTS.

Grease extractor. F. E. LYNN (U.S.P. 1,679,728, 7.8.28. Appl., 9.6.26).—A condensing chamber for solvent is situated above the extraction chamber containing the material, and the connexion between the two is through a siphon which operates when sufficient liquid solvent has collected in the condensing chamber. The solvent plus grease passes through a restricted outlet to a distilling chamber, which is heated and the vapour returned to the condensing chamber.

B. M. VENABLES.

Manufacture of margarine and similar butter-like edible fats. L. N. REDDIE. From A.-G. F. MEDIZ. PROD. CHEM. FABR. (B.P. 295,884, 6.12.27).—By extraction of vegetable or animal oils (e.g., egg-yolk oil) with hot alcohol a mixture of lecithin, sterols, and oil or fat is obtained, which is readily emulsifiable with water. The extract is mixed with edible fats to form margarines.

E. LEWKOWITSCH.

Manufacture of highly sulphonated oils of vegetable or animal origin. ERBA A.-G. (B.P. 292,574, 15.12.27. Ger., 23.6.27).—Vegetable or animal oils are sulphonated in the presence of substances (e.g., per-salts or hydrogen peroxide) which are capable of evolving active oxygen in acid solution. The product is washed in the usual way or may be neutralised by alkali and the alkali sulphates precipitated by the addition of water-soluble alcohols. E. LEWKOWITSCH.

Centrifuging of wax [from honeycombs]. C. BOESCH (B.P. 287,094, 13.3.28. Ger., 14.3.27).

Emulsions etc. (G.P. 446,162).—See III. Vermicide (B.P. 296,608).—See XVI.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Detection of adulteration of turpentine oil. G. DUPONT and BARRAUD (Bull. Inst. Pin, 1927, 87—90; Chem. Zentr., 1927, II, 2123—2124).—The method is based on the Christiansen phenomenon whereby an opalescent mixture of two immiscible liquids each having the same n value for a given spectrum colour and temperature and thereby appearing homogeneous produces an alteration in spectrum colour and indices of refraction on alteration of temperature. Glycerin of known refractive index is mixed with the turpentine oil and the temperature is measured when the emergent colour from the dark rim of the bright surface changes from indigo to violet. The point where the refractive index curves of the two pure liquids intersect gives for a definite temperature a definite colour to the emergent light. E. H. SHARPLES.

Zinc oxide. DANIELS.—See VII.

PATENTS.

Catalytic production of metal resinates etc. [driers]. J. CHABANNAIS (F.P. 625,219, 29.11.26).—Resins, fatty acids, or glycerides are heated at 150—200° with steam under pressure and the metal oxide is slowly added with constant stirring. The process avoids the necessity of using precipitated hydroxides, the steam acting as a catalyst in converting the oxide into resinate, oleate, stearate, etc. A. R. POWELL.

Manufacture of oil colours and impregnation materials from petroleum pitch. HABA G.M.B.H. FÜR INDUSTR. BETEILIGUNG, Assees. of R. WEITHÖNER (G.P. 447,470, 27.2.25).—Petroleum pitch is heated at 300° with lime, zinc oxide, manganese oxide, lead oxide, or alumina, and linseed oil. A. R. POWELL.

Phenol resin compositions. BAKELITE CORP. (B.P. 280,520, 27.10.27. U.S., 10.11.26).—A phenol is condensed with excess of formaldehyde in presence of a basic catalyst (sodium carbonate). The resin so produced, containing no free phenol and therefore non-darkening, is further condensed with urea in the presence of an acid catalyst (lactic, boric, or phosphoric acid), the excess of formaldehyde being thereby absorbed.

S. S. WOOLF.

Manufacture of condensation products of arylamines. P. HALLER and H. KAPPELER (B.P. 274,501, 16.7.27. Switz., 16.7.26. Addn. to B.P. 266,358; B., 1928, 224).—The condensation products described in the earlier patent are treated with an organic base (methylamine, pyridine), a neutral or acid salt (sodium bisulphite), or an organic acid (acetic acid, tannic acid).

S. S. WOOLF.

Manufacture of combined pieces, particularly sheets or plates of artificial resins. Soc. CHEM. IND. IN BASLE (B.P. 286,250, 27.2.28. Switz., 1.3.27).—Compound plates are formed by joining sheets of urea-formaldehyde resins at any intermediate stage of their

production, and curing as a whole. Suitable cement may be used if desired. "Non-splinter" sheets are made by using inner layers of softer material than that composing the outer layers. S. S. WOOLF.

Process of sticking, cementing, impregnating, or filling, and manufacture of agents therefor. CONSORT. F. ELEKTROCHEM. IND. G.M.B.H. (B.P. 271,090, 11.5.27. Ger., 11.5.26).—Polymerisation products of vinyl acetate soluble in alcohol, benzene, etc. are claimed. S. S. WOOLF.

Polymerised hydrocarbons (U.S.P. 1,679,093). **Emulsions etc.** (G.P. 446,162).—See III.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Purification and fractionation of caoutchouc. VII. R. PUMMERER, A. ANDRIESSEN, and W. GÜNDEL (Ber., 1928, 61, [B], 1583—1591; cf. A., 1927, 1193).—The behaviour of different preparations of caoutchouc towards continuous extraction with flowing ether has been examined. "Total caoutchouc" is dissolved to the extent of 65—70% in about 40 hrs.; at this point an inflexion of the time-amount dissolved graph occurs and dissolution subsequently proceeds very slowly. The residual "gel caoutchouc" appears approximately homogeneous. Caoutchouc derived from latex preserved with ammonia and subjected to dialysis followed by coagulation and extraction with acetone, is much more slowly soluble in ether and the inflexion occurs after much more prolonged extraction. A very similar graph is obtained for the dissolution of Para crêpe, which is soluble to the extent of about 52%. The same crêpe, after extraction with acetone and subsequent preservation under carbon dioxide to prevent disintegration by the solvent, dissolves comparatively very readily in ether and leaves about 16.8% of gel skeleton. If, however, preservation is considerably prolonged, the residue amounts to 28.1% of the original material, indicating a re-formation of the gel caoutchouc originally present. The effect of the acetone is mainly attributable to the temperature employed in the extraction. The graph for "smoked sheet" rises steeply and shows a fairly well-defined inflexion at 70% after 60—70 hrs.; the end-point is reached after 700 hrs., the sol caoutchouc amounting to 86%. The high content of the latter is due to the exposure of the crude caoutchouc to a temperature of 40—50° during smoking. The initial fractions are intensely orange coloured, but the colourless third fraction affords pure caoutchouc practically free from nitrogen. The subsequent fractions contain a small but increasing proportion of nitrogen. The gel skeleton appears to contain about 20.4% of protein. The numerical relationship of "sol" to "gel" caoutchouc must be determined in each sample; an equilibrium exists which can be attained from either side. Gel formation is regarded as a process of orientation of colloid-chemical or possibly crystallographical nature due to extra-molecular forces. It is considered most probably as an association and not as polymerisation due to main valencies. H. WREN.

Preparation and mol. wt. of isocaoutchouc-nitron. VIII. R. PUMMERER and W. GÜNDEL (Ber., 1928, 61, [B], 1591—1596).—*iso*Caoutchouc-nitron is

most readily prepared by the action of nitrosobenzene on caoutchouc dissolved in the minimum quantity of cold benzene in an atmosphere of nitrogen or carbon dioxide. The solution rapidly loses viscosity and the reaction is complete in 1—2 days, after which the solution is poured in a thin stream into an excess of light petroleum (b.p. 40°). The product is thus obtained in pale yellow flocks which do not exhibit crystalline structure. It dissolves fairly readily and completely in cold benzene, but the solubility diminishes when it is preserved, probably owing to aggregation. It undergoes auto-oxidation much more readily than does caoutchouc. Determinations of mol. wt. in freezing benzene or nitrobenzene give values lying between 1200 and 1400 provided that measurements are not made until the properties of the solutions have become constant. This value corresponds well with a parent caoutchouc molecule of 8 isoprene groups which have reacted with 8 mols. of nitrosobenzene with loss of 16 atoms of hydrogen. The data thus agree closely with the lowest values observed for caoutchouc in menthol.

H. WREN.

Constitution of caoutchouc. H. LOEWEN (Kautschuk, 1928, 4, 163—164, 194—195).—A call is made for further investigation of the chemical constitution of caoutchouc as distinct from physical theories as to its structure. Volatile and crystallisable products of probable value for further elucidation of the constitution of caoutchouc have been obtained by the action of water on the ozonisation product of a chloroform solution of acetone-extracted crêpe rubber which had been treated previously with approximately one half of the theoretical proportion of hydrogen chloride.

D. F. TWISS.

Plasticity and elasticity of rubber. A. VAN ROSSEM and H. VAN DER MEYDEN (India-rubber J., 1928, 76, 360—363).—Compression-recovery tests indicate that masticated rubber at 16° does not show real plasticity, but only pseudo-plastic flow with an elastic after-effect, whereas at 70° real plasticity is observed without appreciable elastic after-effect. With vulcanised rubber also the three properties of instantaneous elastic recovery, slow elastic after-effect, and permanent plastic deformation can be discerned; for under-vulcanised rubber (92: 71) the first two are less marked than the last, but with increasing degree of vulcanisation the elastic recovery increases, and at 18° may attain 100%, but at 147° the maximum observed was 65%, the elastic after-effect and plasticity being 7% and 28%, respectively.

D. F. TWISS.

Thermoplastic products resembling gutta-percha and shellac, from rubber. F. KIRCHHOF (Kautschuk, 1928, 4, 142—149).—The thermoplastic products derived from rubber by the action of sulphuric acid, organic sulphonic acids or their chlorides (cf. Fisher, B., 1928, 61) are in principle identical with those described earlier by the author (e.g., B., 1921, 91 A), whose experiments have also included rubber latex. The resinous African rubbers differ from *Hevea* rubber in that with sulphuric acid they can yield a shellac-like product. The designation "thermoprene" and "thermoplastic substance" for these products is unsatisfactory, and an alternative classification is suggested for the more important types

of thermoplastic polypropylenes or *cyclocàoutchoucs* based on their method of formation. D. F. TWISS.

Abrasion tests of rubber stocks containing various types of carbon black. W. B. PLUMMER and D. J. BEAVER (Ind. Eng. Chem., 1928, 20, 895—899).—Using the type of abrasion-testing machine described by Williams (B., 1927, 635), it is found that the abrasion loss per unit of work done increases with increase in the size of the abrasive up to 0.45 mm. and then decreases rapidly; this may explain frequent discrepancies between laboratory and road tests. Comparison of an ink-making black, lamp black, thermatomic black, acetylene black, and two gas blacks in vulcanised rubber shows the greatest abrasion-resistance to be given by a gas black. Ageing at 70° causes a marked decrease in resistance to abrasion; the percentage degradation is not uniform, however, and is generally higher for the mixings of greater abrasion resistance. After ageing, indeed, the relative position of two mixings containing different blacks may be reversed. D. F. TWISS.

Evaluation of variable-temperature cures [in the vulcanisation of rubber]. J. R. SHEPPARD and W. B. WIEGAND (Ind. Eng. Chem., 1928, 20, 953—959).—It is generally difficult to foretell with any exactitude the relative effect of different "stepped cures," i.e., of different vulcanisation processes effected with variable temperature. Assuming that the intensity of curing or vulcanisation action is doubled for each rise of 8.3°, it is possible to evaluate mathematically the total effect of a vulcanisation process occurring over a range of steadily varying temperature. Formulae and graphs are given to facilitate such calculations. D. F. TWISS.

Carbon black. JOHNSON.—See II.

PATENTS.

Vulcanisation of [rubber] latex. I. G. FARBEREND. A.-G. (B.P. 288,206, 2.4.28. Ger., 4.4.27).—Latex, containing an aromatic sulphonic acid or sulphonate of the type obtained by the condensation of naphthalene, formaldehyde, and sulphuric acid (cf. B.P. 7138; B., 1914, 366), or a sulphonated mineral oil, aliphatic tar oil, naphthenic or fatty acid, or a fat splitter as dispersion medium, is vulcanised by treatment with sulphur dioxide and hydrogen sulphide at ordinary temperature.

D. F. TWISS.

Vulcanising natural or artificial rubber. METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 291,438, 12.5.28. Ger., 2.6.27).—Products with high resistance to abrasion are obtained by vulcanising rubber in the presence of organic compounds formed from hydrogen selenide, ammonia, and aldehydes, with or without the additional presence of other known accelerators.

D. F. TWISS.

Manufacture of rubber and the like. P. SCHIMROWITZ, Assr. to VULTEX, LTD. (U.S.P. 1,682,857, 4.9.28. Appl., 14.3.23. U.K., 14.9.22).—See B.P. 208,235; B., 1924, 142.

Comminuting rubber (B.P. 295,435).—See I.

XV.—LEATHER; GLUE.

Depilation of skins by means of alkaline solutions. R. H. MARRIOTT (J. Soc. Leather Trades' Chem., 1928, 12, 281—303; cf. B., 1928, 580).—Alkaline solu-

tions containing reducing agents, e.g., sodium sulphite-sodium sulphide, can only loosen the hair on skins if the final liquor has $p_H > 11$. The effect of the hydroxyl-ion concentration on hair is very complicated. The nitrogen-containing fraction hydrolysed by a soak liquor at about p_H 10 differs from that split off at p_H 9.0—9.5 since the loss of the nitrogen fraction over this range inhibits the subsequent action of the sodium sulphide. Cystine, especially in a reduced form, is fairly rapidly decomposed by lime suspensions giving rise to ammonia, and it appears to affect the decomposition of gelatin, but only in the early stages of the reaction. The addition of sodium sulphide, though reducing cystine, inhibits the decomposition of the amino-acid. The presence of gelatin tends to preserve sodium sulphide. Tests with other alkalis on cystine show the following order of diminishing effect in hydrolysing cystine:—lime water and lime suspensions, hydroxides of barium, strontium, sodium, potassium, and magnesium, and sodium carbonate. This is also the order of diminishing depilatory powers. Lime is more effective in breaking down cystine and in producing active reducing agents than are the other alkalis. Lime suspensions do not produce much ammonia from gelatin, urea, glycine, or aspartic acid. Gelatin yields less ammonia than egg or blood albumin when treated with lime liquor. More ammonia is obtained from coagulated egg albumin than from uncoagulated. Coagulation liberates sulphhydryl groups. Experiments with collagen, hair, collagen and hair, collagen and cystine, and cystine, respectively, with lime suspensions, also collagen and sodium sulphide, show that the ammonia content rises rapidly at first and then the rate of increase becomes less with increasing time. More ammonia is produced from hair than from collagen. Sodium sulphide produces less ammonia from collagen than does lime, and less ammonia is produced from collagen when hair is present. Probably lime and sodium sulphide attack the protein molecule in different ways. It does not necessarily follow that lime has a more vigorous action on collagen.

D. WOODROFFE.

Permeability of hide and leather. II. **Permeability to gases.** M. BERGMANN and S. LUDEWIG (Collegium, 1928, 343—351; cf. B., 1928, 205).—Measurements have been made of the volume of gas forced through various leathers; the experimental error was 1%. As the gas permeated the leather the latter absorbed up to 9% of moisture in 100 hrs. and the permeability fell by 53%, showing that the greater the moisture content of the leather the lower is the permeability to gases. The permeability to gases is independent of the gas pressure and practically independent of the position of the leather (grain or flesh side). The diminishing order of permeability of a number of samples of leather is as follows:—alum-tanned goatskin, chrome-tanned sheep, sumach-tanned sheep, chrome-tanned goatskin, chrome-tanned calfskin, iron-tanned leather, crust-tanned hide, finished hide, long-process pit-tanned hide. Patent leather was quite impermeable to gases.

D. WOODROFFE.

Electrically determining the penetrability of sole leather for water. J. N. GERSSEN (Collegium, 1928, 337—343).—An improvement of Jablonski's apparatus

(*ibid.*, 1925, 616), in which a copper plate is in contact with the under-side of the leather and a small electric current is passed when the water has just penetrated; this current is increased by means of a transformer and made to give automatic notification. D. WOODROFFE.

Tanning materials of the British Empire. IV. (Bull. Imp. Inst., 1928, 26, 311—322).—A number of lesser-known tanning materials are dealt with, which are not at present in commerce but appear worthy of consideration. A bibliography and an index to the series are included.

Formaldehyde as tanning material. B. A. TARACHOVSKI (Vestnik, 1925, [2], 102—103).—The skins (100 pts.) are placed in a closed drum with water (80 pts.) and a slightly alkaline formaldehyde solution (10 per 100 pts.); the concentration of formaldehyde during tanning is 0.5—3.5%. CHEMICAL ABSTRACTS.

[Tannins in] pine bark. P. YAKIMOV and B. ANIKIN (Vestnik, 1926 [8-9], 68—79).—Exposure to rain or transportation of logs by water causes little direct loss of tannin, but storage in the damp state causes loss of tannin through mould growth. The total soluble matter, but not the ratio of tans to non-tans, decreases with increasing age and size of the tree. CHEMICAL ABSTRACTS.

Density of vegetable tannins and of the skin. D. SHURAVLIEV and D. ARBUSOV (Vestnik, 1926, [12], 41).—The densities of tannins vary with the source. Collagen has $d_{4} 1.42$. CHEMICAL ABSTRACTS.

Absorption of vegetable tannins by hide powder under different conditions. G. W. SCHILLER and B. SCHWARTZBERG (Vestnik, 1926, [1], 45—47).—Addition of acid increases both total and "irreversible" fixation of tannin; sulphitation decreases the former, but not the latter. Fixation increases with increase of the ratio of tannin to hide powder (for constant volume), and is independent of this ratio when the concentration of the solution is constant. CHEMICAL ABSTRACTS.

Tanning process. P. I. PAVLOVICH (Vestnik, 1926, [10—11], 60—65).—The maximum tannin fixation observed by Thomas and Kelley at p_H 7—8 is ascribed to subsequent washing of the hide powder with water at p_H 5.8. A procedure therefore employs pretanning at p_H 7—8, and re-tanning at the natural p_H value of the extract (about p_H 5.5). CHEMICAL ABSTRACTS.

Dry chrome tanning. N. KOTELNIKOV and I. BASS (Vestnik, 1926, [2—3], 45—47).—A procedure is described. CHEMICAL ABSTRACTS.

PATENTS.

Impregnation of leather. J. J. J. GUILLEMIN (B.P. 272,197, 26.5.27. Fr., 2.6.26).—The product used is prepared by mixing 20 pts. of fossil or vegetable resins or artificial gum at 150—300° with 45 pts. of a gum-dissolving oil, *e.g.*, linseed oil, at 130—150°, allowing the mixture to cool to 70—80°, and stirring in a mixture of 1 pt. of litharge in 34 pts. of turpentine or "white spirit." D. WOODROFFE.

Drying of leather. V. G. WALSH (B.P. 295,804, 7.7.27).—Vegetable-tanned leather is drummed with a

non-volatile, non-injurious, acid substance having a high absorptive capacity for the colour of the tan, *e.g.*, a solution of a vegetable tannin saturated with alum, to which has been added sufficient alkali to precipitate some aluminium hydroxide, with or without addition of borate, glucose, and gum gattau. The leather is then dried in an atmosphere containing naphthalene. D. WOODROFFE.

Preparation of depilatory substances. R. BOTSON, ASSR. to SOC. INDUSTRIELLE DES APPL. CHIM., SOC. ANON. (U.S.P. 1,683,442, 4.9.28. Appl., 19.8.26. Belg., 21.8.25).—See B.P. 285,152; B., 1928, 342.

Method of slicking hides. W. C. WRIGHT (B.P. 296,219, 27.10.27).

XVI.—AGRICULTURE.

Potassium xanthate as a soil fumigant. E. R. DEONG and J. TYLER (Ind. Eng. Chem., 1928, 20, 912—916; cf. B., 1926, 207).—Experiments are described in which potassium xanthate was used against beetles and the root-knot nematode (*Caconema radiculicola*). The decomposition of xanthate by acids was more rapid when the theoretical amount was supplied as hydrochloric acid than when calcium superphosphate was used, more than 48 hrs. being required in the former case. The penetration of carbon disulphide from xanthate buried in soil with superphosphate amounted to 6.18 in. laterally and 5 in. downward in subsoils after 3—5 days. Wet soils absorb more carbon disulphide than dry ones, and clays more than sands. The % mortality—time curves of xanthate used alone or in conjunction with superphosphate or sulphur invariably showed a steady fall until about the fifth day, followed by a slight rise for the subsequent 5—6 days, and thence a final steady decline. In field experiments autumn fumigation was more efficient than in winter or early spring. Deep ploughing-in of a well incorporated mixture of xanthate (200—400 lb.), superphosphate (200—400 lb.), and sulphur (100 lb. per acre) is recommended. A. G. POLLARD.

Nitrification in Hungarian Szik (alkali) soils. F. ZUCKER (Z. Pflanz. Düng., 1928, 12A, 102—107).—Nitrification in Szik soils is an exceptional occurrence, and is confined to the upper layers. The determining factors are not as yet fully understood.

H. J. G. HINES.

Does the supply of root-soluble nutrients in manured and unmanured soils alter during a growing period? H. NEUBAUER, W. BONIEWITZ, and A. SCHOTTMÜLLER (Z. Pflanz. Düng., 1928, 12A, 108—114).—Phosphoric acid and potassium available to plants, as determined by the seedling method, are not altered during the normal period of vegetative growth. Nine tenths of the potassium added as chloride or as dung was taken up by the seedlings. The uptake of phosphoric acid from superphosphate and basic slag was about three tenths of the added quantity.

H. J. G. HINES.

Nutrient action of iodine in Chile sodium nitrate [dressings for sugar beet]. H. ELLEDER (Z. Pflanz. Düng., 1928, 12A, 97—101).—In a field experiment with sugar beet little difference between synthetic and

Chilean sodium nitrate could be detected. Further additions of potassium iodide in conjunction with heavy nitrogenous manuring depressed the yield of roots and increased the yield of leaves. H. J. G. HINES.

Carbohydrate changes during ripening of plantains. S. RANGANATHAN, JUN. (J. Indian Inst. Sci., 1928, 11A, 80—83).—No diastatic activity in ripening plantains was observed, and no inhibitive matter could be extracted from the pulp. Oxygen seems essential to the ripening process. A. G. POLLARD.

Influence of soil reaction on results obtained by the Neubauer method. B. DIRKS (Z. Pflanz. Düng., 1928, 12A, 65—96).—Potassium chloride extracts of zeolitic and non-zeolitic soils when titrated with lime water show but little difference in the titration curves. On boiling, however, the curves of zeolitic soils flatten between the range p_H 8.0—8.3. The length of this flat portion of the curve is a measure of the amount of zeolites in the soil. Values of p_H obtained on the unheated extract give little guide to the lime requirement. On the boiled extract the value 8.3 is taken as the critical p_H , and further additions of lime would prove injurious. Part of this injurious action is ascribed to soluble calcium bicarbonate. The Neubauer results are not influenced by reaction so long as the p_H of the boiled extract does not exceed 8.3. The easily soluble calcium bicarbonate can be removed by heating the soil on a water-bath, and soils so treated give better results by the Neubauer method. H. J. G. HINES.

Determination of the lime requirement of soils. GAY [with P. MÜLLER and O. ROOS] (Z. Pflanz. Düng., 1928, 7B, 393—434).—The various existing methods for determining lime requirement of soils are compared from the point of view of their limitations and the differences between experimental results and those of field trials. Results of numerous comparative tests are recorded and a form of group classification is adopted. The best representation of the "lime condition" of a soil is obtained from a consideration of the analytical figures for chalk, exchangeable calcium, and hydrolytic and exchange acidities. All four values are necessary in order completely to specify the soil condition. Determinations of p_H values, though not necessary, serve to amplify the above results when used in conjunction with the titrimetric acidity determinations. The *Azotobacter* reaction in its present form is not trustworthy. A. G. POLLARD.

Determination of carbon dioxide in carbonates in soil. A. RIAD (Analyst, 1928, 53, 486—487).—Hepburn's modification of the Van Slyke method of determining carbon dioxide in carbonates (B., 1927, 42) has been successfully applied to soils. Precautions should be taken to minimise the period of contact between the baryta and atmospheric air, and a pressure reduced to 6—7 cm. of mercury instead of 2 cm. will suffice. D. G. HEWER.

Determination of iodine in soils. R. V. NORRIS and D. A. R. RAO (J. Indian Inst. Sci., 1928, 11A, 75—79).—The method of Hercus, Benson, and Carter (J. Hygiene, 1924, 24, 321) does not yield absolute results for iodine in soils, the presence of much ferric oxide leading to loss of iodine. The method adopted is

as follows:—A nickel boat containing at least 5 g. of the soil is placed in a combustion tube inside a silica electric furnace and heated at 850° in a stream of oxygen. The end of the combustion tube is drawn out to a capillary which enters the absorption vessel containing 10% sodium hydroxide solution. After heating for 1 hr. the alkali solution is neutralised with sulphuric acid, 2 c.c. of carbon tetrachloride are added, and the iodine is liberated by the addition of 3—5 drops of nitro-sulphuric acid (nitrous fumes from treatment of starch with nitric acid absorbed in sulphuric acid). The carbon tetrachloride solution is separated, washed twice with very small quantities of water, treated with 2 c.c. of sodium acetate solution, and titrated with N/1270-sodium thiosulphate solution. By this method 5 p.p.m. of iodine in soil can be detected and larger amounts determined. A. G. POLLARD.

Methods of preparation of soil for mechanical analysis. A. N. PURI and B. M. AMIN (Agric. Res. Inst. Pusa, Bull. No. 175, 1928, 15 pp.).—In using the Robinson pipette method for mechanical analysis of soils (cf. B., 1922, 990 A), disturbance of the suspension during sampling is minimised by bending the tip of the pipette at right angles to its length and by using an automatic suction apparatus. Mutual interference of particles during settling occurs in suspensions of clay soils of concentrations greater than 1%. Tables are provided whereby the effect of temperature on settling velocity can be corrected. In the preliminary treatment of soil for analysis, treatment with hydrochloric acid is necessary; 0.05N-acid (cold) suffices for this purpose, and the use of hydrogen peroxide (English official method) or of hot 0.2N-hydrochloric acid (Hissink) is unnecessary and too drastic. A. G. POLLARD.

PATENTS.

Root-filling substance. E. G. HEDSTRÖM (U.S.P. 1,678,940, 31.7.28. Appl., 13.11.26. Swed., 27.9.26).—A natural asphalt, preferably Syrian asphalt, is dissolved in benzol, chloroform, or other solvent, which is then partially evaporated. The addition of bismuth salts, or a noble metal in colloidal condition, enables the position of the filling to be detected by Röntgen rays. F. G. CLARKE.

Preparation of an [earth] worm-killing composition. PREMIER OIL EXTRACTING MILLS, LTD., and W. E. WATERHOUSE (B.P. 296,608, 19.12.27).—Mowrah seed, cake, or meal is extracted with hot water under raised pressure, and the extract is filtered and evaporated to dryness under reduced pressure. L. A. COLES.

XVII.—SUGARS; STARCHES; GUMS.

Adsorption properties of bone char. T. B. WAYNE (Ind. Eng. Chem., 1928, 20, 933—940).—Sugar solutions were tested for ash and invert sugar content before and after filtration through various samples of bone char, of which the analyses and p_H values are given. Char carbon, freed from ash by acid treatment and washing, was found to adsorb no ash. If heated or made alkaline with lime some adsorption occurs. The adsorptive power of char ash is high. With increasing alkalinity of the char ash adsorption is

increased up to a point. A similar effect is obtained by increasing the alkalinity of the syrup. Invert sugar disappears to a greater extent with the more alkaline chars. Chars containing calcium carbonate and underburned were found to lower the p_H value, probably owing to hydrolytic adsorption. The effect of increasing the proportion of char to liquor was to increase ash adsorption, but the p_H value was unchanged. This indicates the probability of exchange adsorption reactions. The effect of change of temperature was also studied.

C. IRWIN.

PATENT.

Removal of hydrochloric acid from sugar solutions. F. KOCH, ASST. to INTERNAT. SUGAR & ALCOHOL Co., LTD. (U.S.P. 1,678,819, 31.7.28. Appl., 9.5.25. Ger., 15.5.24).—The sugar solution is sprayed against a hot stream of fluid, e.g., oil or air, and is subsequently separated therefrom. The addition of sulphuric acid facilitates the process. F. G. CLARKE.

XVIII.—FERMENTATION INDUSTRIES.

Wines from grapes attacked by *Eudemis*. II. J. H. FABRE and E. BRÉMOND (Ann. Falsif., 1928, 21, 323—339; cf. B., 1928, 104).—The action of alkali carbonates and calcium carbonate as neutralising agents for these infected wines has been investigated with a view of obtaining data which will enable the fraudulent neutralisation of such wines to be detected. Results of analyses of several wines of this type of different origin, and also of the difference in taste of the wines at different periods of time after treatment with these carbonates, are tabulated. By comparing the density and the analytical values obtained for fixed acidity, total ash, soluble ash, total tartaric acid, and total alkali and calcium with those given in the tables, the neutralising agent which has been used can be detected.

T. M. A. TUDHOPE.

Determination of manganese in Peloro [Messina] wines. (SIGNA.) G. SOLARINO (Boll. Chim. farm., 1928, 67, 481—483).—The dry ash from 100 c.c. of the wine is taken up in concentrated nitric acid, the solution being diluted with water, treated with about 0.5 g. of lead dioxide, boiled, allowed to settle, and titrated with 0.01N-oxalic acid to determine the permanganic acid formed; 0.1 c.c. of 0.1N-oxalic acid corresponds with 0.00012 g. Mn. In 20 wines, containing between 12.2 and 14% of alcohol by vol., the manganese content varied from 0.0011 to 0.0024 g. per litre.

T. H. POPE.

Character in pot still whisky. II. S. H. HASTIE and W. D. DICK (J. Inst. Brew., 1928, 34, 477—490; cf. B., 1926, 604).—The presence in whisky of furfuraldehyde, which is one of the chief character-giving substances, is ascribed to its production towards the end of the first distillation by the local superheating or partial charring of the pentoses in the presence of the acids contained in the wash. In the second distillation the shape of the still head and lyne arm have a limited effect, the transfer of furfuraldehyde being controlled mainly by the temperature of distillation corresponding to a more or less definite strength of distillate. When the alcoholic content of the charge of

the spirit still is in practice 20% by vol., the maximum transfer takes place at 201° F. with the strength of the distillate approx. 50% of alcohol by vol.

C. RANKEN.

Importance of hydrogen-ion concentration for the distillery. W. DIEMAIR and K. SICHERT (Biochem. Z., 1928, 198, 1—18).—The hydrogen-ion concentration of raw and stewed potatoes, of acid and ripe yeast, and of the mash at different stages of fermentation is determined, and titration curves are constructed. For the determination of buffering power the hydrogen-ion concentration and titratable acidity, determined against azolitmin, phenolphthalein, and neutral-red, are obtained. In the raw and stewed potato extracts, formol-titratable and total soluble nitrogen are determined. The hydrogen-ion concentration, titratable acidity, and buffering power increase during the mashing process. By bringing the degree of acidity of acid yeast to 1.0, the acidity is regulated for the mash and an optimum produced for diastase (p_H 4.8—5.0). The behaviour of mash during fermentation may be characterised by the following three stages: (a) (24 hrs.) chief fermentation in which hydrogen-ion concentration and titratable acidity increase, complex proteins are split up, and organic acids and primary phosphate are formed; (b) (48 hrs.) the p_H value and titratable acidity remain practically constant and fermentation slows off; (c) (72 hrs.) a slight increase of hydrogen-ion concentration, a decomposition of formed hydroxy-acids, and a slight decrease of titratable acidity.

P. W. CLUTTERBUCK.

Anti-ferments. HARVEY.—See XX.

PATENTS.

Manufacture of yeast. ZELLSTOFF-FABR. WALDHOF, and O. LÜHRS (B.P. 287,052, 6.3.28. Ger., 12.3.27).—Substances containing sugar are fermented and the yeast is collected for withdrawal at the centre of the bottom of a vat by imparting to the lower layer of the liquid a rotatory or gyratory movement which is limited by baffles to the lowermost zone of the vat. The gyratory movement may be produced by the admission of mash or other liquid at the bottom of the vat.

C. RANKEN.

Medicinal products obtained from yeast. I. S. MACLEAN (B.P. 295,757, 23.5.27).—An activated medical product is obtained by incubating yeast in a solution containing phosphates and carbohydrates and subjecting the resulting sterols or sterols and fats to ultra-violet light.

C. RANKEN.

Urease preparation. O. LIND (U.S.P. 1,679,250, 31.7.28. Appl., 3.9.27. Ger., 15.9.26).—Soya beans, or other vegetables containing urease, are extracted with water to which alkali phosphates are added before or after the extraction, and the separated extract is dried by atomising into a dry and heated gas stream.

F. R. ENNOS.

Production of an alcoholic stimulant. T. D. KELLY (B.P. 295,822, 29.7.27).—A mixture of fermented materials, such as ethyl alcohol, with a small quantity of glycerol is subjected to the action of ultra-violet rays, an alkaline substance, soluble in the liquid, being added to counteract any tendency towards acidity. The

stimulant may be solidified by the addition of substances of a colloidal or farinaceous nature, raising the temperature to 50—80°, and finally cooling suddenly.

C. RANKEN.

XIX.—FOODS.

Influence of temperature on rate of corrosion of copper by milk. G. N. QUAM, E. I. SOLOMAN, and A. HELLWIG (Ind. Eng. Chem., 1928, 20, 930—931).—The solubility of copper in milk, the time of contact being 30 min., increases with rise in temperature to a maximum at 85—90° and decreases with further rise. The break in the curve is probably due to decreasing solubility of oxygen in the milk. The disagreement with the observations of Rice and Miscall (J. Dairy Sci., 1928, 1, 320), who found that the solubility of copper in milk at b.p. was less than at room temperature, is due to the use of a longer time of contact by these workers, whereby the change in composition of the milk at the higher temperature became an important factor.

W. J. BOYD.

Textures of ice creams as influenced by some constituents. M. H. GIVEN (Ind. Eng. Chem., 1928, 20, 966—968).—The addition of gelatin (0.3%), raw eggs (2.5%), or dry egg yolk (0.3%) to the ice-cream mix increases the viscosity of the mix and improves the texture of the final product. Gelatin (0.5%) gives a product which is almost gummy and therefore objectionable. The use of 22% of evaporated milk in place of 18% of market milk and 4% of cream gives a product similar in viscosity and texture to that made with 0.3% of gelatin.

W. J. BOYD.

Water in cream. A. F. LERRIGO (Analyst, 1928, 53, 488—489).—Out of 21 samples bought as cream or preserved cream, not sterilised or clotted, 19 contained less than 8.0% solids-not-fat in serum, 18 less than 7.5%, 13 less than 6.5%, 8 less than 5.5%, and 2 less than 4.5%, the cream having been thinned with water.

D. G. HEWER.

Determination of added phenol and cresol in milk. H. T. FAWNS (Analyst, 1928, 53, 489—490).—Added phenol and cresol may be detected in milk by an application of Mumford's method (cf. B., 1913, 671). To 50 or 25 c.c. of milk 2 c.c. of concentrated sulphuric acid are added to precipitate casein and sulphonate the phenolic compound. The precipitate is collected, and filtrate and washings are heated at 85° for 10 min., cooled, and the coagulated albumin is removed by filtration. To the filtrate 5 c.c. of 10% potassium nitrate solution are added, nitration is effected at 80—90°, and the liquid filtered if necessary, cooled, and made alkaline with ammonia solution (*d* 0.880). A yellow coloration due to ammonium picrate (which can be determined colorimetrically) indicates the presence of phenol. The reaction is quantitative for additions of phenol of 0.1—0.005 g. per 50 c.c. of milk.

D. G. HEWER.

Effect of dry skim milk on baking quality of various flours. E. GREWE (Cereal Chem., 1928, 5, 242—255).—Besides increasing the nutritive value of bread the addition of dried skim milk (4%) to the dough gives a decided improvement in the baking quality of certain wheat flours. Flours differ in their

reaction to the use of dried skim milk in bread making. In some the baking qualities are improved, and in others they are not. Hard spring-wheat flours react more favourably than hard winter-wheat flours. The range of fermentation time in which doughs give good bread is increased by the use of dried skim milk. The lactose present imparts a good colour to the crust, and the general texture of the bread is improved.

T. M. A. TUDHOPE.

Applications of spectrophotometric methods to baking problems. E. L. STEPHENS, A. M. CHILD, and C. H. BAILEY (Cereal Chem., 1928, 5, 256—268).—The colour of baked products containing flour and molasses was examined spectroscopically, and the percentage reflection of light measured between 500 and 620 mμ. The colour is influenced to a small extent by the colour of the molasses used, and to a greater extent by the amount of sodium bicarbonate present; the colour darkens with increase of alkalinity and with increase of temperature of baking. T. M. A. TUDHOPE.

Chemical changes accompanying the western yellow blight of tomato. J. T. ROSA (Plant Physiol., 1927, 2, 163—169).—The total nitrogen, alcohol-insoluble nitrogen, and diastatic activity of the leaves decrease, whilst the reducing sugars, sucrose, starch, and alcohol-soluble nitrogen increase. Carbohydrates accumulate and total and alcohol-soluble nitrogen increase in the stems and roots, whilst diastatic activity disappears. The inception of blight appears to be connected with inability to translocate nitrogen and to synthesise higher nitrogen compounds.

CHEMICAL ABSTRACTS.

Vitamin-B content of avocados. L. S. WEATHERBY and E. W. WATERMAN (Ind. Eng. Chem., 1928, 20, 968—970).—Feeding experiments on rats showed that the fresh pulp of avocados has about one twelfth the vitamin-B content of dry yeast.

W. J. BOYD.

Fumigation tests with ethylene dichloride-carbon tetrachloride mixture [on foodstuffs etc.]. L. F. HOYT (Ind. Eng. Chem., 1928, 20, 931—932).—A mixture of 3 pts. of ethylene dichloride and 1 pt. of carbon tetrachloride was used in the proportion of 7 lb. per 500 cub. ft. for the fumigation of cereals and food products infested with *Tribolium* and Indian meal moth. Complete control of the insects was obtained. After fumigation for 24 hrs. the germinative power of wheat, oats, peanuts, and clover was practically unaffected, whilst that of beans was considerably reduced. Unwrapped foodstuffs, especially those rich in fat, absorbed considerable amounts of the fumigant, but were edible after 1—8 days. Bread baked from fumigated flour was indistinguishable in appearance and taste from the controls. A similar concentration of fumigant gave a 100% kill of house-moth larvae in furniture.

A. G. POLLARD.

Tests for incipient rancidity of fats. DAVIES.—See XII.

PATENTS.

Treatment of wheat. L. E. SMITH, Assr. to INTERNAT. MILLING Co. (U.S.P. 1,679,099, 31.7.28. Appl., 23.5.25).—The rapid ageing of wheat is brought about by allowing it to flow by gravity in a thin sheet through a vertical

tank, which contains a series of hopper cones below each of which is an inverted, perforated, distributor cone. The wheat is subjected to independently regulated currents of moist air from pipes placed below each distributor cone. F. R. ENNOS.

Preservation of eggs. A. H. PENFOLD (B.P. 296,012, 23.5.27).—Liquid egg or egg yolks are mixed with 10–20% of dextrose, cooled to -26° , and kept at about this temperature during storage. F. R. ENNOS.

Treatment of liquids such as milk etc. with rays, particularly ultra-violet rays. G. HOFFMANN (B.P. 279,902, 31.10.27. Aust., 30.10.26).—The milk is caused to trickle in a thin layer down over a cylinder of wire gauze or other grid-like structure within which is the source of radiation. B. M. VENABLES.

[Spreading device for] drying apparatus for maize, tea, barley, wheat, etc. T. CLARKE and DAVIDSON & Co., LTD. (B.P. 296,221, 29.10.27).

Drying of hay, straw, etc. C. LYON (B.P. 296,162, 4.7.27).

Preserving food (U.S.P. 1,680,934).—See V. Margarine etc. (B.P. 295,884).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Presence of *o*-arsanilic acid in technical arsanilic acid. N. G. POZDNIakov (J. Chem. Ind., Moscow, 1928, 5, 530).—Technical arsanilic acid contained over 4% of the *o*-isomeride. This is seven times as soluble in water as the *p*-compound, and advantage is taken of this difference in solubility for the purification of the latter. R. TRUSZKOWSKI.

The genus *Mentha*. XVI. **Non-volatile constituents of *Mentha aquatica*, L.** S. M. GORDON (Amer. J. Pharm., 1928, 100, 433–449, 509–524).—The dried ground leaves (13.78 kg.) of *M. aquatica*, L., were extracted with alcohol and a black extract (2.6 kg.) was obtained. Steam-distillation of the extract gave 108 g. of volatile oil [d_4^{20} 0.8649, n_D^{20} 1.4588, acid value nil, ester value 39.79, ester value after acetylation 120.5, ester (as linalyl acetate) 13.93%, alcohol (as linalol) 36.43%] containing furfuraldehyde and sesquiterpenes. The aqueous distillate contained a relatively large amount of acetic acid and a small quantity of trimethylamine. The residue from the distillation consisted of a dark aqueous liquid containing potassium nitrate (58 g.); formic acid; a yellow pigment (0.08 g.), m.p. 250–253°; dextrose (3 g.); succinic acid; rhamnose; betaine (4.1 g.); choline; a glucosidic material; and a black oily resin from which the following substances were isolated: methylamine; ammonium chloride; linalol (34 g.), obtained by steam-distillation after alkaline hydrolysis; dotriacontane (3 g.), m.p. 69.5–70°; a phytosterol, m.p. 133.5–134°, presumably α -sitosterol; 66 g. of an oil boiling over a large range and yielding pronounced sterol colour tests; lupeol, m.p. 210°; a substance ($C_6H_{12}O$)_x, m.p. 212°; "aquaticol" (6 g.), m.p. 273°, possibly identical with micromerol; a compound, $C_{26}H_{42}O_3$, m.p. 261°; a compound $C_{22}H_{38}O_3$, m.p. 258°; a compound, m.p. 319°; and 644 g. of fatty acids consisting of butyric, hexoic, heptoic, linoleic, linolenic, oleic, palmitic, and possibly stearic, melissic

(m.p. 86.5°), and myristic acids, and an acid, $C_{25}H_{50}O_2$ (?), m.p. 82–83°. E. H. SHARPLES.

Constituents of Indian essential oils. XXIV. **Essential oil from rhizomes of *Curcuma zedoaria*, Roscoe.** B. S. RAO, V. P. SHINTRE, and J. L. SIMONSEN (J.S.C.I., 1928, 47, 171–172 T).—Distillation in steam of the rhizomes of *C. zedoaria* yielded 0.94% of oil, of which approximately one third was heavier than water; it had d_4^{20} 0.9724, n_D^{20} 1.5002, acid value 1.3, saponif. value 3.0, saponif. value after acetylation 66.6. The oil was shown to have the following composition: *d*- α -pinene 1.5%, *d*-camphene 3.5%, cineole 9.6%, *d*-camphor 4.2%, *d*-borneol 1.5%, unidentified alcohols trace, sesquiterpenes 10.0%, sesquiterpene alcohols 48%, residue (probably mainly sesquiterpene alcohols) 21%.

Russian essential oils. A. TSCHERNUCHIN (Masloboino-Shirowoje Djelo, 1927, [3–4], 57–60; Chem. Zentr., 1927, II, 2122).—The best yield of oil containing the maximum quantity of menthol is obtained from *Mentha piperita* by using the half-blossomed plants. Plants in full bloom give the smallest yield of oil and the lowest menthol content. For the purification of the oil it is shaken with freshly precipitated aluminium hydroxide, and after keeping for 3–5 hrs. is filtered.

E. H. SHARPLES.

Essential oils as anti-ferments. E. H. HARVEY (Amer. J. Pharm., 1928, 100, 524–529).—The anti-ferment efficiency of 32 typical essential oils has been measured by methods similar to those previously described (B., 1924, 845). Acetic acid is an efficient anti-ferment at concentration above 1.5%. Exposure of essential oils to ultra-violet light lowers their anti-ferment efficiency. E. H. SHARPLES.

PATENTS.

Manufacture of organic arsenic compounds. G. NEWBERRY, and MAY & BAKER, LTD. (B.P. 295,744, 23.3.27).—*o*-Acylaminophenolarsinic acids are condensed with chloroacetic acid (or its esters or amide) to give the corresponding aryloxyacetic acids, which on removal of the acyl group are converted into benzo-*p*-oxazine derivatives. Thus, 3-acetamido-4-hydroxybenzenearsinic acid and chloroacetic acid yield 3-acetamido-4-carboxymethoxybenzenearsinic acid, from which by hydrolysis with alkali 3-hydroxybenzo-*p*-oxazine-6-arsinic acid is obtained; chloroacetamide gives 3-acetamido-4-carbamylmethoxybenzenearsinic acid and the same oxazine. 3:5-Diacetamido-4-carboxymethoxybenzenearsinic acid and 8-amino-3-hydroxybenzo-*p*-oxazine-6-arsinic acid are similarly prepared. C. HOLLINS.

Manufacture of aliphatic auromercaptocarbonylic acids. CHEM. FABR. AUF ACTIEN, FORM. E. SCHERING (B.P. 266,346, 15.2.27. Ger., 17.2.26. Addn. to B.P. 265,777; B., 1927, 349).—A β -thiol-aliphatic acid (e.g., cysteine hydrochloride reduced with sulphurous acid) is treated with an auric salt (potassium bromaurate). C. HOLLINS.

Manufacture of complex [organic] metallic compounds. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 295,734, 17.3.27).—An alkali salt of pyrocatechol mono- or di-sulphonic acid, pyrogalloldi-sulphonic acid, or protocatechuic acid is treated with the

hydroxide, oxide, or carbonate of a metal (excluding alkali and alkaline-earth metals, and also excluding antimony and bismuth with all except pyrogalloldisulphonic acid), alkali or an amine being added to give a neutral complex salt. The additional alkali is unnecessary when cadmium is the metal used. Copper, arsenic, cadmium, manganic, stannous, vanadyl, aluminium, ferric, antimony, bismuth, and lead complex salts are described (cf. B.P. 213,285; B., 1925, 691).

C. HOLLINS.

Manufacture of metal salts of sulphoarsenol. F. LEHNHOFF-WYLD (U.S.P. 1,674,711, 26.6.28. Appl., 9.11.25).—See B.P. 232,612; B., 1925, 970.

Medicinal products (B.P. 295,757). **Alcoholic stimulant** (B.P. 295,822).—See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Chemical development and fixation of latent finger prints. G. POPP (Z. angew. Chem., 1928, 41, 1005—1007).—The iodine method for fixing finger prints on documents, letters, etc. is difficult of application. The process recommended is to fix the iodine-print with a 0.001% solution of palladium chloride. After removing excess of the reagent and drying slowly, the print appears in a deep brown marking. Alum or tannin may be added to the reagent to prevent the effacement of the print by water-soluble inks. In some cases the paper ground becomes tinted by the iodine, and the use of palladium chloride is inadvisable. The print may be fixed with dilute ammonia solution.

A. G. POLLARD.

PATENT.

Production of photographic plates, films, etc. for indirect three-colour photography. E. A. LAGE (U.S.P. 1,683,560, 4.9.28. Appl., 13.3.26. Ger., 23.10.25).—See B.P. 266,468; B., 1927, 349.

XXII.—EXPLOSIVES; MATCHES.

Explosive properties of hypochlorites. WEICHERTZ.—See VII.

PATENT.

[Granulation of] explosives. O. MATTER (B.P. 296,600, 25.11.27).—The explosive is dissolved in a solvent and the solution is added with stirring to a liquid that mixes in all proportions with the solvent, but does not dissolve the explosive. Thus, pentaerythritol tetranitrate and tetranitromethylaniline are dissolved in acetone and the solution is poured into water maintained continuously in motion.

S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Pollution of tidal and non-tidal waters. J. H. COSTE (J.S.C.I., 1928, 47, 133—139 t).—The general effect of pollution of all kinds is to degrade the normal life of affected streams so that a river may become unable to support fish life, or become a nuisance or even a

danger to health. The ecology of polluted rivers has been studied to some extent, and three types of association have been observed, characterised respectively by the water shrimp *Gammarus*, the *Sialis* group, and by tube-forming worms and midge larvæ, the last-named group usually occurring in the blackmud of badly-polluted waters, whilst *Gammarus* and snails are associated with pure water and form a large part of the food of fishes. The one point common to all organic pollution is that the polluting matters need oxidation. Apart from oxygen liberated by photo-synthesis the only source of this element is the air. The amount of oxygen dissolved by water from the air is very small—only about 1/100,000 of the weight, or 7/1000 of the volume of the water at 15°. The rate at which this can be taken up is also very limited. When polluting matter enters a tidal reach, it will travel upwards, so that a long stretch of water above and below is affected, whilst the final escape of pollution is much retarded. Although these effects of tide are unfavourable to the rapid removal of pollution, the continual disturbance of the river bottom prevents the accumulation of foul mud and the movement of the water is favourable. The chief causes of unfitness of water for supporting fish life are:—(1) deficiency or great excess of oxygen; (2) presence of specifically toxic substances or of fine suspensions which will foul the gills of fish; (3) absence of necessary salts; (4) an unsuitable temperature. It appears that of all fish the *Salmonidae* need the most oxygen, and eels and other bottom-living fish the least. The high requirements of salmon account for their abandonment of rivers which have, by reason of pollution, become depleted of dissolved oxygen. It is probably useless to expect salmon and their allies to frequent waters containing less than 70% or other fish less than 50% of saturation of this element. Pollution of rivers has, to some extent, been restrained, or sought to be restrained, by enactment. The earlier Acts only dealt with the matter from the point of view of public health, but in 1923 the Salmon and Freshwater Fisheries Act, which affords some protection to fish, was passed. The question of standards of purity to be applied to rivers is considered. Whether they are based on analytical figures or on behaviour, they should be in such form that they may easily be understood. It is not necessary that they should be unvarying, but changes should be infrequent and tactfully introduced. The economic necessity of any specific form of pollution, and the possibility of recovery of useful products, should be considered before distinctly purificatory treatment is adopted. Sewage should offer little real difficulty, but industrial effluents need a full understanding of the features of each case. The system adopted by the Ministry of Fisheries of interesting local people in river surveys, and of arousing civic and æsthetic consciousness, is more likely to remedy the evils of pollution than premature resort to legal proceedings.

PATENT.

Treatment of water. J. W. M. BOURGOGNION and C. L. PHILIPS (U.S.P. 1,683,521, 4.9.28. Appl., 17.5.27. Holl., 10.5.26).—See B.P. 271,079; B., 1928, 770.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

NOV. 9, 1928.



L.—GENERAL; PLANT; MACHINERY.

Hot centrifuge. C. HÜTTER (Chem. Fabr., 1928, 543—544).—A centrifuge for use in the separation of the constituents of a mineral mixture by gravity in a fused salt mixture of high sp. gr. comprises four small wire-wound vertical tube furnaces evenly disposed around the axis of the ordinary laboratory centrifuge. Electrical contact with the heating current is made by means of brushes running on two contact rings below the rotating stage carrying the furnaces. The temperature is regulated by means of an external variable resistance and the centrifuge is driven by means of a small direct-drive motor in the foot of the shaft. To carry out a test a mixture of 0.5—3 g. of the mineral mixture is mixed with the powdered salt and introduced into a small silica or hard glass tube. When the mass has fused the centrifuge is started and run for 5 min. at 2000 r.p.m. After cooling, the tube is cut in half and the salt mixture dissolved from each separately. A. R. POWELL.

New sampling pipette for sampling viscous liquids and lard-like substances. L. ALLEN (Chem. Umschau, 1928, 35, 228).—The sampler consists of a hinged double scoop which is plunged open into the material to be tested; the leaves are then closed by a sliding collar and thus a cylindrical sample can be withdrawn representing all layers. E. LEWKOWITSCH.

Waste heat recovery. W. GREGSON (Proc. Inst. Mech. Eng., 1928, 169—236).

Utilisation of waste heat for steam raising. G. MARTIN (Trans. Ceram. Soc., 1928, 27, 222—241).

See also A., Oct., 1086, **Adsorption of mixtures of gases** (KLOSKY and WOO). 1101, "Area" of contact catalysts (CONSTABLE). 1105, **Sampling granular material** (BAULE and BENEDETTI-PICHLER). **Radio-metric microanalysis** (EHRENBERG). 1106, **Electro-metric titrations** (RABINOVITSCH and KARGIN). **Micro-analysis** (BERMEJO Y VIDA). 1009, **Acid-alkalimeter** (GOODE).

Flash-point apparatus. MEYERHEIM and FRANK.—See II.

PATENTS.

Retort-furnace. C. V. MCINTIRE, Assr. to CONSOLIDATED COAL PRODUCTS Co. (U.S.P. 1,681,946, 28.8.28. Appl., 15.7.24).—Transverse flues are formed within a horizontal retort by means of inverted, trough-shaped, metal sections which constitute a lining for the bottom of the retort. Hot gases are passed through the flues thus formed between the lining and the refractory retort bottom, grooves in which engage the outer edges of each metal section. F. G. CLARKE.

Kilns. CARBORUNDUM Co., LTD. From CARBORUNDUM Co. (B.P. 297,241, 12.11.27).—In a kiln as described in B.P. 266,140 (B., 1927, 351) the products of combustion are exhausted through ports evenly distributed over the floor of the work chamber. B. M. VENABLES.

Heat-exchange apparatus. C. F. BRAUN (U.S.P. 1,683,236, 4.9.28. Appl., 31.8.26).—A multi-tube and shell-type heat interchanger is arranged so that both fluids flow longitudinally and the fluid in the tube makes three passes for each one of the outer fluid, two passes being countercurrent and one concurrent. B. M. VENABLES.

Pulverising machine. E. T. ROEBKE, Assr. to AMER. PULVERIZER Co. (U.S.P. 1,683,304, 4.9.28. Appl., 12.3.27).—The rotatable hammer heads of a disintegrator comprise alternate discs and spaced bars arranged in arcs of equal outer diameter. B. M. VENABLES.

Pulverising machine. A. M. READ (U.S.P. 1,683,627, 11.9.28. Appl., 31.12.19. Renewed 28.2.24).—A cylindrical barrel is approximately horizontal, but has its lower side inclined away from the feed end. It is provided with balls or other grinding media, and with at least one perforated transverse screen dividing it into at least two compartments. The perforated screens are concave to the feed end. B. M. VENABLES.

Ball-mills [for pulverising coal and feeding it to boiler furnaces]. P. L. CROWE (B.P. [A] 296,682, [B] 296,697, 1.3.27).—The apparatus described in U.S.P. 1,627,487—8 (B., 1927, 511) is in (A) provided with a ring of pole pieces attached to the mill itself, which form part of the electric motor for driving. In (B) several mills are combined in series with different sized balls in each stage. B. M. VENABLES.

Uniform admixture of finely-powdered material. SOC. ANON. DES CEMENTS LUXEMBOURGEOIS (Swiss P. 120,750, 28.5.26).—The material is stirred while suspended in about 50% of its volume of air, e.g., in a cylindrical vessel with a conical base provided with inlet tubes supplying air to replace that escaping from the mixture. L. A. COLES.

Subliming apparatus. H. G. STONE (U.S.P. 1,683,931, 11.9.28. Appl., 2.6.22).—A heated plate with low walls at the edge has a conveying means running over it, and chambers above for collecting vapours which discharge into condensers sideways over the edge walls. B. M. VENABLES.

Emulsifier. W. THORDARSON (U.S.P. 1,683,500, 4.9.28. Appl., 13.4.27).—A wheel with flat radial paddles revolves within a casing having sharp-edged corrugations on its interior surface and provided with an inlet at the top and outlet at the bottom. B. M. VENABLES.

Rotary agitators. A. E. WHITE. From PFAUDLER Co. (B.P. 296,247—8, 9.1.28).—A propeller is constructed of stamped sheet metal or tubes having the same thickness everywhere, and hence very suitable for coating with vitreous enamels.
B. M. VENABLES.

Feeding materials into high-pressure vessels. I. G. FARBENIND. A.-G. (B.P. 274,122, 11.7.27. Ger., 10.7.26).—An air-lock device is provided with an outlet valve (to the main reaction vessel) having a large opening, whilst the other closure or inlet valve is formed as a piston which when depressed forces the material into the main chamber and at the same time displaces nearly all the high-pressure gas out of the air-lock chamber. The piston is not withdrawn until after the outlet valve is closed.
B. M. VENABLES.

Filters or the like. C. S. GARLAND (B.P. 296,359, 30.5. and 7.6.27).—The preliminary filtering or straining material of the filter comprises helically woven wire or strip the apertures of which are a maximum when the wires are at right angles, but when the mesh is stretched in either direction the apertures become diamond-shaped and smaller. The strainer preferably takes the form of a tube supported between end discs, the distance apart of which can be varied. Within the strainer may be situated a finer filter, preferably a pile of discs forming an edge-filter, or loose filling may be inserted.
B. M. VENABLES.

Apparatus for making solutions [from materials partly insoluble]. H. FRISCHER (B.P. 281,695, 1.12.27. Ger., 6.12.26).—The material is charged to the interior of a perforated drum attached to the stirring device in a dissolving vessel, the stirring blades project beyond the drum, and the latter retains the undissolved matter.
B. M. VENABLES.

Determining the concentration of turbid suspensions. R. E. HALL, Assrs. to J. M. HOPWOOD (U.S.P. 1,681,339, 21.8.28. Appl., 13.12.24).—A measured quantity of the suspension is diluted until a luminous object attains a predetermined standard of visibility, when viewed through a predetermined thickness of the diluted suspension. From the quantity of the latter, the original concentration is determined.
F. G. CLARKE.

Apparatus for concentrating solutions. FELD & VORSTMAN G.M.B.H. (G.P. 449,216, 5.8.24).—The liquor compartment contains below the heating tubes one or more depositing surfaces of such shape that deposited material slides down them by gravity, e.g., two superposed cones. These are connected with the heating chamber by a tube through which condensed water passes.
L. A. COLES.

Preventing separation of the constituents of liquid dispersed systems. ANODE RUBBER CO., LTD. From P. KLEIN, A. SZEGVÁRI, S. GOTLEB, and G. F. WILSON (B.P. 296,454, 3.3.27).—In order to maintain uniformity of composition in liquid dispersions or emulsions, e.g., natural or artificial dispersions of unvulcanised or vulcanised rubber, which are liable to coagulation by agitation or shock, the fluid is kept in constant upward movement by withdrawal near the surface and reintroduction at the bottom; the rotary

impelling device is so constructed as to minimise the shocks between it and the dispersion and to produce the desired rate of flow with a minimum speed of revolution.

D. F. TWISS.

Apparatus for effecting reactions between gases and liquids, especially for the production of sulphuric acid using nitric acid or oxides of nitrogen. J. FISCHLER (G.P. 449,286, 10.3.26).—The liquid is atomised upwards by jets in a gas tube situated in the lower part of the reaction chamber, through circular devices corresponding to the atomising cone, which also serves for the withdrawal of the gases from the apparatus.
L. A. COLES.

Separating and liquefying gaseous mixtures, especially air, into the components. C. W. P. HEYLANDT (B.P. 271,520, 23.5.27. U.S., 24.5.26).—Compressed air (or other mixture of gases) at atmospheric pressure is divided, one portion being expanded in an engine and the other through a throttle valve; the first portion passes through part only of a heat exchanger attached to a double-column liquefier, and the second part passes through the whole of the heat exchanger. The heat exchanger is cooled by the dissolved nitrogen.
B. M. VENABLES.

Removal of dust from hot gases containing dust and oil particles. TROCKNUNGS-, VERSCHWELUNGS-, & VERGASUNGS-GES.M.B.H. (G.P. 449,185, 3.6.25).—The gases are treated with a counter-current of steam superheated to a temperature above the dew point of the oil.
L. A. COLES.

Fractional condensation of mixed vapours. L. WEISBERG, Assr. to BARRETT Co. (U.S.P. 1,676,232, 3.7.28. Appl., 29.11.21. Renewed 23.3.27).—In a fractionating column the total reflux is divided at a suitable point, one portion being utilised as reflux for fractional condensation of the rising vapours.
C. HOLLINS.

Furnace [draft] regulation. SMOOT ENGINEERING CORP., Assces. of C. H. SMOOT (B.P. 282,437, 16.12.27. U.S., 16.12.26).

Furnace arches. H. W. SPENCER. From F. B. BIGELOW (B.P. 297,271, 17.1.28).

[Floors for] muffle furnaces. GIBBONS BROS., LTD., and T. E. BRIDGFORD (B.P. 296,936, 4.1.28).

[Portable] apparatus for producing heat or coldness by means of chemical reactions. K. GYÖRGY (B.P. 285,511, 18.2.28).

Refrigerating apparatus. T. ZEMANN (B.P. 289,088, 23.4.28. Austr., 22.4.27).

[Bearings for sugar-cane] crushing machines. D. B. COOPER (B.P. 297,283, 22.2.28).

Melting furnace (B.P. 282,627).—See X.

Precipitation from gases (B.P. 296,956).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Coking coals and the mechanism of the coking process. P. DAMM (Brennstoff-Chem., 1928, 9, 293—295, and Stahl u. Eisen, 1928, 48, 1330—1332).—Either the "oily-bitumen" content (cf. Fischer, B., 1925, 233) or the caking index as determined by Meurice's method

gives a satisfactory measure of the caking power of a coal. A distinction is drawn between the "swelling" of a coal and the "swelling pressure," the former being the volume increase on coking when the coal is free to expand, and the latter the pressure developed when the coal is coked in a confined space. No definite relationship exists between caking power and swelling pressure, although a good coking coal must develop a certain pressure on coking as well as possess a suitable caking index. A knowledge of the amount of volatile matter evolved before, during, and after the plastic period throws considerable light on the mechanism of the coking process and on the behaviour of particular coals.

A. B. MANNING.

Chemical changes occurring in the coking and oxidation of a bituminous coal. O. KÜNLE (Brennstoff-Chem., 1928, 9, 295—298).—The influence of the temperature of coking on the yield and composition of the coke from a Saxon coal has been investigated by the use of the crucible method for the determination of volatile matter. The amount of volatile matter evolved rose from 24.9% at 400—450° to 47.8% at 1000—1020°. At temperatures below about 350° considerable oxidation occurred. After 10 hrs. heating at 225° the oxygen content of the coal had risen from 9.9% to 23.8%. The oxygen content of a sample of fusain, separated from the coal and heated under the same conditions, rose from 5.5 to 17.4%.

A. B. MANNING.

Relation between humic acid and lignin. W. FUCHS (Brennstoff-Chem., 1928, 9, 298—302).—The physical and chemical properties of the nitration products of a humic acid from brown coal and samples of lignin of different origin show them to be compounds of closely related constitution. From a study of their composition, behaviour on methylation, mol. wt. of the methylated products, etc. it is concluded that they are isonitrosoketohydroxycarboxylic acids of a radical of high mol. wt. and probably of cyclic structure. The principal difference between the lignin and the humic acid derivatives lies in the higher nitrogen content of the former. This may be due either to the presence of double linkings in the lignin molecule, or to its possession of more hydroxyl and less carboxyl groups than the humic acid, so that its more pronounced phenolic character would bring about a certain amount of true aromatic nitration.

A. B. MANNING.

Improvement of the calorific value of coke-oven gas. R. SCHÖNFELDER, W. RIESE, and W. KLEMP (Ber. Ges. Kohlentechn., 1927, 2, 250—258; Chem. Zentr., 1927, II, 2251).—The gas is passed successively over catalytic material at 700° comprising nickel precipitated on magnesite to destroy organic sulphur compounds and to break down heavy hydrocarbons, and, after removal of hydrogen sulphide, over pieces of clay containing reduced nickel at 420° to reduce carbon monoxide and carbon dioxide to methane and water by means of the hydrogen in the gas. The calorific value is raised, e.g., from 4136 to 5368 thermal units.

L. A. COLES.

Explosive gas mixtures. P. H. PRAUSNITZ (Z. angew. Chem., 1928, 41, 1066—1069).—Tests on mixtures

of coal gas, hydrogen, or petrol vapour with air or oxygen have shown that a porous quartz plate sealed into the combustion tube efficiently prevents back-firing when the composition of the gas mixture is within the explosion limits, provided that the plate does not become red hot. The fineness of the pores necessary depends on the nature of the gas mixture; for coal gas-air mixtures glass plates with medium-sized pores suffice, but for oxygen-hydrogen mixtures silica plates with fine pores are required. Back-firing may, however, take place behind the plate if the gases are supplied to the mixing chamber under very different pressures.

A. R. POWELL.

Desulphurisation of lignite oils. B. SALADINI (Annali Chim. Appl., 1928, 18, 337—352).—The decrease in sulphur content and the loss of oil have been determined when a crude lignite oil containing 1.4% S was subjected to the action of various reagents. The following treatments were adopted, the figures in brackets giving the final percentage sulphur content: 5% of sulphuric acid, *d* 1.30 (1.1); *d* 1.84 (0.65); 5% of fuming sulphuric acid (0.25); sodium hypochlorite solution (0.75); sodium hypochlorite solution followed by filtration through alumina (0.55); action of 10% of acetyl chloride and 2% of phosphoric oxide for 10 hrs. (0.8); 10% of anhydrous zinc chloride (1.10); reduction by 10% of stannous chloride until the evolution of hydrogen sulphide ceased (0.62); reduction by 10% of iron dust and 10% of hydrochloric acid (1.30); boiling for 3 hrs. with anhydrous aluminium chloride, 2.5% AlCl₃ (0.52); 20% AlCl₃ (0.08); sodium hydroxide (1.10); boiling for 6 hrs. with 10% of sodium plumbite (0.95); boiling with (a) cupric oxide (1.3), (b) reduced copper (0.8), (c) litharge (0.83), (d) mercury (1.3), (e) sodium (0.92), and (f) sodium amalgam (0.68); with lead soap containing sodium carbonate (0.33). The vaporised oil was passed at 500° over the following reagents: granular copper oxide (1.10); a mixture of 75% of copper oxide, 10% of litharge, 15% of ferrous oxide (0.9); a mixture of equal parts of manganese dioxide, soda-lime, and quicklime (0.86); anhydrous cuprous chloride (0.8); pumice ignited after soaking in copper chloride and sodium plumbite solutions (0.64). When the loss of oil during these treatments is considered in conjunction with their relative efficiencies it is concluded that the most promising reagents are sulphuric acid, aluminium chloride, and lead soap.

F. G. TRYHORN.

Discharge-stability tests on insulating oils. W. ZIMMERMANN (Petroleum, 1928, 24, 1213—1230).—Tests have been carried out on the oils by means of the S.S.W. apparatus, which differs from previously described apparatus in that a fixed potential difference with a variable electrode gap is used. By including a circuit-breaker in the low-tension circuit of the 110—220/30,000 volt transformer, immediate cessation of the discharge is effected following each individual test, thus protecting both the oil and the electrodes; otherwise, a prolonged discharge after the breakdown potential gradient is reached causes a lowering of the discharge stability ("breakdown voltage"). Individual figures for the discharge stability obtained from successive discharges using the same sample of oil show great variation.

The minimum value is usually given by one of the earlier of a series of discharges, often by the first, but may be given by even the tenth. When the first discharge gives the lowest value of the discharge potential this is not necessarily due to incomplete cleansing of the electrodes. The minimum value may vary by as much as 50% below the mean of a series of determinations: it is questionable whether such a mean value alone is adequate for characterising an oil. In a long series of discharges (100—150) there is a tendency for the discharge stability to rise until the final value for the mean figures reaches the maximum obtainable by thorough drying. The steepness of the curve for the average figure gives an indication of the ease of desiccation of the oil, or of its original moisture content. Exchange of moisture between the oil sample and the atmosphere has a great influence on the discharge stability. Careful sampling of the oil is necessary since different samples of oil from the same consignment show great variation in discharge stability. The usual commercial spindle oil and benzene have the same discharge stability as insulating oils; petroleum is twice as stable. Used oils may be brought up to their original insulating value by refining by means of flordin alone or with acid.

W. S. NORRIS.

Testing of transformer oils. I. MUSATTI and R. VOLTERRA (Giorn. Chim. Ind. Appl., 1928, 10, 397—408).—Previous work (B., 1926, 65) is continued, the oxidation of transformer oils being studied in order to ascertain a sufficiently rapid and exact means of classifying these oils with regard to their ageing during use. The method of oxidation in air at 112° in copper cylinders is modified in various ways. All methods based on oxidation of the oil in the open are long and inconvenient and are liable to error, due to loss of the products. Investigations have been made on a rapid method of oxidation out of contact with air and in a closed vessel either in presence of oxygen or with a supply of oxygen to maintain a constant pressure in the oxidation chamber, and in absence or presence of copper. The results obtained show that the most suitable temperature is 200° and that the test should last for 2—3 hrs. The pressure should be kept constant, especially when the oxidation is protracted. The nature of the oxidation is essentially the same in presence and in absence of copper, but the accelerating influence of the metal is intensified under constant pressure. Oxidation of the oil in the air in copper cylinders gives, as regards tendency to formation of sludge, results similar to those furnished by oxidation in oxygen, but the latter method allows of a better differentiation of the oils and leads to greater development of acidity. No relation is apparent between the results of these oxidation methods and those given by the "life test" as used in America. The latter gives a measure of the resistance of an oil to the initial formation of sludge, and the oxygen method a measure of the velocity with which sludge-formation and development of acidity proceed.

T. H. POPE.

Flash-point determinations [for oils] in the normal flash-point apparatus. G. MEYERHEIM and F. FRANK (Petroleum, 1928, 24, 1235—1237).—Closely agreeing values for the flash points of thirteen lubricating and insulating oils are obtained using the following

flash-point apparatus: (a) Marcusson's apparatus with porcelain, Krupp's V2A steel, or brass crucible; (b) the Schlüter-Göckel apparatus with 2 mm. clearance between the thermometer bulb and the side or bottom of the crucible; (c) the Bochow-Sommer and Runge apparatus, clearance 2 mm. or 5 mm., this clearance being accurately reproducible by reason of the thermometer socket and crucible top being mechanically positioned relatively to the crucible. W. S. NORRIS.

See also A., Oct., 1086, **Charcoal and its adsorption** (OKAZAWA). 1088, **Crystalline graphite** (CORDEBAS). 1099, **Detonation-wave in gaseous mixtures** (PAYMAN). **Striae in explosion waves** (CAMPBELL and FINCH). 1130, **Phenols from alkaline solution** (VAVON and ZAHARIA).

Phenols of coal tar. BRÜCKNER.—See III. **Determination of carbon monoxide and methane.** KELLER and KLEMP.—See VII.

PATENTS.

Production of artificial fuel. II. E. WETHERBEE, R. F. GRANT, and H. M. HANNA, Assees. of H. E. WETHERBEE and W. L. JACOBUS (B.P. 274,046, 24.5.27. U.S., 10.7.26).—Briquettes are formed from anthracite or other carbonaceous fuel by mixing the finely-divided material with a composite binder, and heating for 2—3 hrs. at about 300°. The binder comprises a filming agent, *e.g.*, kerosene (10%), a coking ingredient, *e.g.*, petroleum pitch of m.p. 57° (45%), and a non-coking toughening agent, *e.g.*, an air-blown petroleum residuum of m.p. 116° (45%).

A. B. MANNING.

Preparation of a fuel product. H. F. MAUREL (U.S.P. 1,682,680, 28.8.28. Appl., 28.4.26).—The fuel is distilled in such a manner that the oil vapours and steam evolved are mixed with the required amount of air, and are circulated through a heating means and then again through the fuel itself.

A. B. MANNING.

Briquetting [of fuel]. P. C. MULLIGAN, Assr. to L. J. HULL and I. M. FOSTER (U.S.P. 1,683,094, 4.9.28. Appl., 4.10.26).—The fuel material is mixed with molasses, the water is expelled by drying, and, after cooling and briquetting, the molasses is carbonised by baking the briquettes.

C. O. HARVEY.

Treatment and formation of artificial fuels. H. E. WETHERBEE, Assr. to R. F. GRANT and H. M. HANNA (U.S.P. 1,683,061, 4.9.28. Appl., 1.10.25).—Moistened anthracite etc., of small irregular size, is treated with a filming oil to displace the water, and is then dried to effect coalescence of the particles.

F. G. CLARKE.

Dehydration of moist fuel. I. G. FARBENIND A.-G. (B.P. 274,858, 18.7.27. Ger., 21.7.26).—The moist fuel is heated at above 200° and under a pressure greater than the corresponding vapour pressure of water, so that the latter is separated in liquid form. Solid fuels are mixed with a suitable liquid fuel before treatment. The process is of especial advantage where the dried fuel is to be hydrogenated.

A. B. MANNING.

Production of carbon. A. LEDERER (B.P. 274,883, 20.7.27. Austr., 21.7.26).—Two different modifications of carbon are produced by the decomposition of carbon

disulphide at 1600—1700° and 1800—1890°, respectively. The former modification (*d* 1·8) is brittle, hard, and black; the latter (*d* 2·3) is soft, grey, and of metallic character. The former may be converted into the latter by heating at above 2800°. The second modification is particularly suitable for the filaments of electric lamps; it can be subjected to an electric stress of 150—250 amp./mm.²

A. B. MANNING.

Apparatus for producing carbon. C. MATLOCK, Assr. to MONROE-LOUISIANA CARBON CO. (U.S.P. 1,681,945, 28.8.28. Appl., 11.6.23).—A hydrocarbon gas and air, insufficient for complete combustion, are agitated in a mixing machine before they are passed to burners in a furnace.

F. G. CLARKE.

Coking retort ovens of the vertical-chamber type. KOPPERS Co., Assees. of J. VAN ACKEREN (B.P. 281,221, 27.5.27. U.S., 27.11.26).—The heating walls of a vertical-chamber coke oven have groups of vertical heating flues, so arranged that there are upper and lower heating zones each having its own burners with air and fuel-gas supply connexions thereto. Each flue consists of a burner-equipped section in series with a section for conveying hot gases, and the flue groups are disposed in the wall with their burner sections located in different zones from those of their hot gas flow-sections and with substantially all the area of the heating wall composed of zones containing burner sections. Each combustion zone in a heating wall has its own outlet for combustion products. A row of reversible regenerators extends alongside the row of chambers and heating walls. The combustion products from half of the heating walls are led into a common duct and conveyed around the chambers into the other heating walls and thence through the out-flow regenerators.

A. B. MANNING.

Vertical-chamber coke-oven batteries. KOPPERS Co., Assees. of J. VAN ACKEREN (B.P. 288,558, 27.5.27. U.S., 12.4.27).—The oven structures of a vertical-chamber coke-oven battery are supported on frames which extend between the structures and the battery foundations and are capable of expansion horizontally, relatively to each of those parts. An air-cooling system, with inlets from and outlets to the atmosphere, helps to reduce the temperature of the frames and the under side of the battery. Beam frames extending side by side below the chambers and heating walls have gas-tight joints between contiguous sides, so that no brickwork is exposed on the under side of the battery. The frames and coke extractor may be directly connected.

A. B. MANNING.

Rotary retort for the drying and low-temperature carbonisation of brown coal etc. DEMAG A.-G. (G.P. 448,667, 26.6.20).—The volatile products evolved in both the drying and the carbonisation zones of the retort are removed by suction. The water vapour etc. evolved up to about 100° is withdrawn through a tube, movable along the axis of the retort, by means of a fan, which is synchronised with the fan removing the volatile products from the carbonisation zone. The position of the tube in the retort is so adjusted that only the volatile products from the drying zone and none of the valuable carbonisation gases are withdrawn through it.

A. B. MANNING.

Carbonising plant and generator for the production of low-temperature tar. C. ERHARD (G.P. 448,535, 3.1.26).—Coals etc. of high water content are carbonised in an apparatus of the type described in G.P. 436,515 (B., 1927, 549), in which the low-pressure steam, which collects in the pressure chamber, is compressed by means of a steam pump into a high-pressure vessel, at the same time being supplied with just sufficient heat to convert it into high-pressure steam of any desired pressure.

A. B. MANNING.

Apparatus for the distillation of carbonaceous materials. P. M. SALERNI, and METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 296,108, 25.2., 12.4., 29.9., and 6.10.27).—A vertical retort contains a number of superposed annular trays, each provided with stirring devices, which bear on the trays mainly by the action of gravity and are so mounted that they are free to move to allow for any distortion which may occur. These stirrers, which are loosely connected to, and are actuated by, a rotary structure surrounding the trays, are so arranged that they direct the material from the outer part of each tray towards the inner part, whence it travels back to the outer part and overflows through a gap into the next tray, until finally discharged from the retort. The trays are heated preferably by conduction from a central heating chamber, the parts of the tray surfaces not in contact with the material under treatment being suitably lagged. The retort has a number of outlets at different levels, each provided with a centrifugal separator for the removal of dust from the volatile carbonisation products.

A. B. MANNING.

Carbonisation and liquefaction of materials of vegetable, animal, or mineral origin. V. CAMBON (F.P. 618,888, 21.11.25).—The undried materials are heated with a definite quantity of water in an autoclave, the liquid products are discharged, and the residue is subjected to further pyrogenetic decomposition in the presence of superheated steam.

A. B. MANNING.

Carburetted processes and apparatus. Soc. ANON. LE CARBONE (B.P. 288,588, 25.5.27. Fr., 13.4.27).—A method of carburisation for internal-combustion engines using a volatile motor spirit consists in decomposing the spirit, mixed with sufficient air for atomisation, by subjecting it to the action of a heated catalyst, and then passing the treated spirit, mixed with the further quantity of air necessary for combustion, to the engine. The catalyst, which consists of a porous active carbon, is heated by the exhaust gases. The spirit may, if desired, be vaporised by passage through heating tubes before treatment with the catalyst.

A. B. MANNING.

Continuous production of concentrated acetate liquor from acid vapours from the carbonisation of wood. VER. F. CHEM. IND. A.-G., Assees. of E. LÖW (G.P. 448,348, 16.2.26).—A portion of the acid vapours is passed through a solution or suspension of a carbonate or hydroxide, whilst the remainder passes to a condensing plant. The pyroligneous acid condensed in the latter is passed into the absorbing solution.

A. B. MANNING.

Water-gas generators for consuming bituminous fuel. PATENT RETORTS, LTD., and T. M. DAVIDSON

(B.P. 296,161, 2.7.27).—A retort, superposed on a water-gas generator, is surrounded by a jacket containing chequered brickwork and communicating through suitably placed apertures with the upper end of the generator. The latter and the chequered brickwork are divided into two sections by vertical partition walls. The hot gases leaving the jacket are used to preheat the blast air. During the blow period the preheated air is passed up through both sections of the generator; steam is then admitted below one of the grates, passes up through the corresponding section of the generator, transversely through the material above the generator partition wall, and thence into the opposite section of the jacket. The operation is then reversed. The gas-making period may be concluded by a short down-run, when steam is admitted at the top of the chequered brickwork jacket and passed down through the generator.

A. B. MANNING.

Generation of [water-]gas. C. W. BOTSFORD, Assr. to W. W. ODELL (U.S.P. 1,682,264, 28.8.28. Appl., 30.8.23).—A water-gas generator is provided with air inlet ports midway between the top and bottom of the fuel bed, which during the blow period is air-blasted alternately up and down. The air is then shut off and a straight up-steam run is made, the steam being introduced from beneath the fuel bed and the gas removed from above the fuel bed.

A. B. MANNING.

Production of generator gas from bituminous coal with by-product recovery. E. FLEISCHER (G.P. 448,333, 21.5.24).—The apparatus comprises a gas producer and a retort so connected that the hot gases from the former, containing as little carbon dioxide and steam as possible, pass directly into the latter at a temperature of at least 900°. Uniform distribution of the hot gases in their passage through the material in the retort is ensured by roof-shaped gas distributors. The issuing gases, when freed from their condensation products, may be mixed with the excess generator gas.

A. B. MANNING.

Apparatus for utilising the heat evolved in gas generators. FRANKFURTER GASGES., F. P. TILLMETZ, and E. SCHUMACHER (B.P. 285,007, 31.1.28. Ger., 8.2.27).—A generator of the type having water-tubes in the walls of the shaft is provided with ordinary straight drums arranged horizontally above and below, to which the tubes are attached. The water-tubes are given a right-angled bend at top and bottom in such a manner that their expansion gives rise to no strain. The upper and lower drums are connected by appropriate return tubes on the outside of the generator. Vertical drums may be employed in place of the horizontal drums, if desired.

A. B. MANNING.

Removal and recovery of heavy hydrocarbons from low-temperature gases. GELSENKIRCHENER BERGWERKS-A.-G., and H. HOCK (G.P. 447,471, 3.11.22).—The gases are treated, under atmospheric or higher pressures and at the ordinary or lower temperatures, with acetone, or solid or liquid mixtures containing acetone. An apparatus suitable for the purpose is described.

A. B. MANNING.

Manufacture of asphaltic, bituminous, or tarry emulsions. SOC. ANON. LA TRINIDAD (B.P. 279,026,

13.6.27. Fr., 15.10.26).—Pulverulent or colloidal mineral substances are mixed with asphalt, bitumen, or tar, so as to form a putty, to which is then added a substantially anhydrous emulsifying agent consisting of a soda or potash soap to which 1—8% of commercial olein has been added. To obtain an emulsion the requisite amount of water is added to the molten mixture.

A. B. MANNING.

Production of purified montan wax. A. SMELKUS (G.P. 447,530 and Addn. G.P. 449,297, [A] 6.11.23, [B] 7.10.25).—(A) The bituminous brown coal, preferably with the addition of a suitable decolorising agent, is extracted with a solvent, e.g., an aliphatic alcohol or ester, which does not appreciably dissolve any of the oxy-acids present in the coal. The material may be treated during extraction, or subsequently, with nascent hydrogen. (B) Activated charcoal may be used as the decolorising agent.

A. B. MANNING.

Oil-cracking still. W. T. and J. W. HANCOCK (U.S.P. 1,680,541, 14.8.28. Appl., 20.9.26).—A rotatable shaft is suitably mounted in an inclined still tube. A number of discs are set rigidly on the shaft, lying in parallel planes and making acute angles with the shaft. Between each pair of discs is set a pair of ball-carrying plates, the plates of each pair being at opposite sides of the shaft. The inner edges of the plates are spaced from the shaft, whereas their outer edges and the peripheries of the discs are slightly spaced from the tube walls. Free balls are located in the spaces between adjacent discs and at opposite sides of the pairs of plates.

W. S. NORRIS.

Conversion of high-boiling into low-boiling hydrocarbons. CARBUROL A.-G. (G.P. 447,755, 2.5.23).—The heavy oil is passed under pressure through a heated tube at a speed of over 1 m./sec. The apparatus is so arranged that the oil, after reaching the most favourable reaction temperature, lying between 400° and 450°, is maintained at that temperature under a pressure of 20 atm. and at the same speed of passage through the tube for 30—80 sec. It is then suddenly permitted to expand.

A. B. MANNING.

Manufacture of liquid hydrocarbons of low b.p. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,034, 22.2.27).—Carbonaceous materials, atomised in a current of superheated steam, are passed through pipes heated to at least red heat, and the resulting gases, containing a high proportion of olefines, are converted into liquid hydrocarbons of low b.p. by the further action of heat, with or without the aid of high pressures or catalysts, and in the absence of materials, e.g., iron or nickel, which give rise to the separation of carbon.

A. B. MANNING.

Production of liquid and gaseous hydrocarbons of low b.p. from mineral and other oils, tars, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 295,974, 21.4.27. Cf. B.P. 294,557; B., 1928, 738).—The material is decomposed by means of a device, e.g., a nickel-chromium rod, which is immersed in the liquid and heated, preferably electrically, to at least red heat, and at the same time is continuously or intermittently subjected to a blast of steam or hydrogen etc.

A. B. MANNING.

Manufacture of low b.p. oils by cracking mineral oils, tars, etc. I. G. FARBERIND. A.-G. (B.P. 272,483, 26.5.27. Ger., 11.6.26).—Oils are cracked by electrically heated bodies, which are submerged in the liquid and kept in motion therein. The process may be carried out in the presence of hydrogen or other gases, and under any convenient pressure. Any carbon which is formed becomes suspended in the liquid and is removed subsequently by filtration. The heating bodies and containing vessels are preferably constructed of materials which, in contact with the oil, do not become charged with electricity of opposite sign to that on the carbon particles.

A. B. MANNING.

Treatment of hydrocarbons. HYDROCARBURES ET DÉRIVÉS (F.P. 618,896, 24.11.25).—A mixture of hydrocarbons is refluxed at 150° with 7–12% of a solid catalyst for about 2 hrs.

A. B. MANNING.

Distillation of petroleum. E. W. ISOM, Assr. to SINCLAIR REFINING CO. (U.S.P. 1,683,135, 4.9.28. Appl., 6.5.24).—Substantially less than a normal charge of oil is introduced into the still and the lighter fractions are withdrawn by way of a refluxing tower down which flows a supply of the crude oil. When the still is full the charge is distilled direct without refluxing.

C. O. HARVEY.

Liquid-phase purification of distillates. T. G. DELBRIDGE and J. B. HILL, Assrs. to ATLANTIC REFINING CO. (U.S.P. 1,682,603, 28.8.28. Appl., 28.6.26).—The condensates from the fractionation of cracked petroleum distillates are brought into contact with fuller's earth at a temperature above their b.p. but under a pressure high enough to maintain them in the liquid state. The products are distilled to separate the lower-boiling fractions from those containing the polymerised oils. The latter may be used as reflux liquid in the fractionation of the distillates.

A. B. MANNING.

Distillation of oil from oil shale. H. B. KIPPER (U.S.P. 1,682,287, 28.8.28. Appl., 2.8.23).—The finely-ground shale is distilled by the passage of a current of hot gases through the mass, which at the same time is mixed and further subdivided by means of a suitable stirring arrangement.

A. B. MANNING.

Treatment [cracking] of [hydrocarbon] oils. H. C. KIRK (U.S.P. 1,676,230, 3.7.28. Appl., 8.12.27).—Carbonisation is avoided by maintaining circulation of the oil by means of streams of oil suitably directed, e.g., alternately upward and downward, the oil mass being carried forward or reversed in direction by a succeeding stream before it has lost the movement imparted by the preceding stream.

C. HOLLINS.

Hydrogenation of cracked hydrocarbon oils. H. ROSTIN (F.P. 603,101, 10.7.25).—The oils are heated with hydrogen prepared by the passage of hydrogen sulphide over silver, which is preferably precipitated in a finely-divided form on asbestos or pumice. The silver sulphide produced is readily reduced again by any gas mixture rich in hydrogen. Treatment of the oils with nascent hydrogen in this manner hydrogenates the unsaturated constituents, converting them into stable oils.

A. B. MANNING.

Prevention of the colouring [and gumming] of

hydrocarbon oils. J. TAUSZ (G.P. 447,557, 17.7.23).—Hydrocarbon oil distillates stable to light and air are obtained by adding to the oils, freed as far as possible from ammonia and amino-compounds, a small quantity of a substance which in an alkaline medium reacts with the oxygen of the air, e.g., hydroxy- or dihydroxy benzene, and/or a small quantity of an acid, e.g., benzoic or stearic acid.

A. B. MANNING.

Refining of [hydrocarbon] oils. L. BURGESS, Assr. to STANDARD OIL DEVELOPMENT CO. (U.S.P. 1,681,395, 21.8.28. Appl., 13.7.23).—The oil is repeatedly washed with 5–10% of fuming sulphuric acid, and, after each washing and separation, is treated with caustic soda and an aliphatic monohydric alcohol to remove the sulphur compounds produced.

F. G. CLARKE.

Treatment of lead sludge [from gasoline purification]. J. B. HILL, Assr. to ATLANTIC REFINING CO. (U.S.P. 1,682,562, 28.8.28. Appl., 17.3.26).—The sludge, separated from the spent aqueous sodium plumbite and dried, is roasted under oxidising conditions to remove sulphur dioxide. It is then dissolved in caustic soda and mixed with the separated spent liquor, for re-use in the purification process.

F. G. CLARKE.

Production of high-viscosity lubricating oil and high-grade asphalt from petroleum. A. E. PEW, JUN., Assr. to SUN OIL CO. (U.S.P. 1,675,462, 3.7.28. Appl., 22.12.26).—Residues of petroleum which has been distilled under non-cracking conditions at low pressure are made to flow in a shallow stream agitated by injection of gas-oil vapours and heated by currents of mercury vapour in tubes wholly immersed in the liquid, which is under highest vacuum obtainable. An extremely viscous lubricating oil and the gas oil are separately condensed, leaving an asphalt containing little insoluble matter.

C. HOLLINS.

Lubricating oils. T. H. SANDERS (B.P. 297,201, 19.8.27).—To overcome "stickiness" of cold lubricating oils when used in internal-combustion engines, over 1% by vol. of an organic ester of low viscosity (amyl acetate, butyl lactate) is added.

F. G. CROSSE.

Apparatus for carbonisation and distillation of wood. R. MALBAY (U.S.P. 1,684,875, 18.9.28. Appl., 21.4.25. Fr., 5.5.24).—See B.P. 233,335; B., 1925, 700.

Dehydration and distillation of tars or oils. D. RIDER, Assr. to THERMAL INDUSTRIAL & CHEMICAL (T.I.C.) RES. CO., LTD. (U.S.P. 1,685,034, 18.9.28. Appl., 28.2.24. U.K., 10.4.23).—See B.P. 221,528; B., 1924, 934.

Plant for washing coal or other mineral substances. E. HODETGE (B.P. 297,007, 9.6.27).

Apparatus for combustion of pulverulent or powdered fuel. J. J. C. BRAND and B. LAING (B.P. 297,240, 11.11.27).

Maintaining the temperature of [fuel] oil within predetermined limits. J. J. KERMODE (B.P. 296,689, 2.5.27).

Apparatus for use in extinguishing burning oil in tanks. ASIATIC PETROLEUM CO., LTD., and A. H. PARKER (B.P. 296,740, 7.6.27).

Lead tetra-alkyl (B.P. 283,913).—See VII. Tiles etc. (B.P. 296,626).—See IX. Filtering material (U.S.P. 1,676,151).—See XXIII.

III.—ORGANIC INTERMEDIATES.

[Separation of the] phenols of coal tar. H. BRÜCKNER (Z. angew. Chem., 1928, 41, 1043—1046, 1062—1066).—The separation of the higher phenols from the crude cresol fraction of coal tar (b.p. < 180°) is best effected by fractional hydrolysis of their sulphonic acids. The mixture is first fractionally distilled, collecting separately the fractions at every 5° between 175° and 215°, fractions above 200° being repeatedly fractionated. Every fraction is then separately heated for several hours at 103° with an equal weight of concentrated sulphuric acid whereby the phenols are converted into their sulphonic acids with a yield of 75—80%. Water is added and the mixture gently distilled in steam at 103—104° to remove unchanged phenols; superheated steam is then passed in and the temperature kept at 116—119° until no further *m*-cresol is obtained, it is then raised to 122—126° whereby phenol is obtained, and finally at 133—136° a mixture of *o*- and *p*-cresols is obtained. In the fractions of the original mixture distilling above 200°, steam distillation of the sulphonic acids yields a small quantity of *o*-4-xenol at 107—110° and of *m*-4-xenol at 120—124°. From the highest-boiling fractions traces of *m*-5-xenol can also be isolated. The mixture of *o*- and *p*-cresol obtained as described above is separated by fusing it with finely-powdered sodium hydroxide and monochloroacetic acid to obtain the corresponding cresoxyacetic acids; the sodium salt of the *p*-acid is only slightly soluble whereas that of the *o*-acid is readily soluble in water. The sulphonic acid method may also be used for the fractionation of technical xenol and is applicable especially to the isolation of the pure *m*-5-compound which alone distils by passing steam through the sulphonic acid mixture at 100°. At 107—111° the *o*-4-isomeride is obtained, at 114—118° a mixture of *o*-3- and *p*-2-, and at 121—125° *m*-4-xenol. The mixture of *o*-3- and *p*-xylenols is separated by taking advantage of the solubility of the sulphonic acid of the former and the insolubility of that of the latter in 50% sulphuric acid.

A. R. POWELL.

See also A., Oct., 1100, Hydrolysis of saccharin (TÄUFEL and others). 1101, Catalysts of acetylation reactions (CONANT and BRAMMANN). 1102, Electrolytic oxidation of organic substances (MARIE and LEJEUNE). Photo-oxidation of organic compounds (PLOTNIKOV). 1106, Micro-determination of sulphur in organic compounds (KUBOTA and HANAI). 1107, Micro-determination of carbon, hydrogen, and nitrogen (BOBRAŃSKI and SUCHARDA). 1112, Catalysts for formation of alcohols from carbon monoxide and hydrogen (FROLICH, FENSKE, and QUIGGLE). 1113, Tests for methyl alcohol (TODD). 1117, Identification of hexamethylenetetramine and formaldehyde (IONESCU). 1125, Hydrogenation of nitrobenzene (VAVON and CRAJCINOVIC). 1127, Catalytic preparation of alkylanilines (ROY). 2:5:6-Trichloro-*m*-4-xylidine and its derivatives (BUREŠ and BORGMANN). 5-Bromo-*m*-4-xylidine (BUREŠ and

MANDEL-BORGMANNOVÁ; WHEELER and THOMAS). 1149, Rapid determination of phenol in glycerol (DENIGÈS). 1155, Toxic derivatives of methane and ethane (HALOFF). Determination of benzene (GADASKIN).

Conductivity of solvents. KEYES and others.—See XI.

PATENTS.

Dehydration of alcohol etc. W. K. LEWIS, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,676,700, 10.7.28. Appl., 16.8.22).—Alcohol is distilled, and the fraction containing over 95—96% of alcohol is redistilled under pressure of 100 lb. or more per sq. in. to remove a constant-boiling mixture and leave anhydrous alcohol.

C. HOLLINS.

Recovery of concentrated acetic acid from dilute acetic acid. H. SUIDA (B.P. 296,172, 18.7.27. Addn. to B.P. 230,447; B., 1925, 827).—Dilute acetic acid is superheated to 150—155° in a vaporiser, and the vapour is passed up a column down which is percolating a solvent which is difficultly soluble in water and has a b.p. above that of acetic acid. Suitable solvents are esters of mono- or poly-basic cyclic carboxylic acids (e.g., *n*-butyl or hexyl phthalate). W. G. CAREY.

Preparation of derivatives of dibenzanthronyl. K. ZAHN and P. OCHWAT, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,675,014, 26.6.28. Appl., 28.12.26. Ger., 30.12.26).—3-Iodo-2-methoxybenzanthrone, m.p. 248°, is heated in nitrobenzene with copper powder to give 2:2'-dimethoxy-3:3'-dibenzanthronyl, m.p. 387—390°, which can be converted by alkaline fusion into a vat dye. (Cf. B.P. 278,047; B., 1927, 903.) C. HOLLINS.

Production of a new acid and its salts strongly absorbing ultra-violet rays. T. SUZUKI and S. SAKURAI, Assrs. to ZAIDAN HOJIN RIKAGAKU KENKYUJO (U.S.P. 1,684,562, 18.9.28. Appl., 11.8.24. Japan, 20.12.23).—See B.P. 242,721; B., 1926, 76.

Manufacture of chlorides of aromatic *o*-hydroxycarboxylic acids. E. B. HIGGINS, Assrs. to BRIT. SYNTHETICS, LTD. (U.S.P. 1,684,273, 11.9.28. Appl., 31.3.27. U.K., 20.7.26).—See B.P. 278,463; B., 1927, 903.

Manufacture of perylene. C. H. MARSCHALK (U.S.P. 1,684,738, 18.9.28. Appl., 1.12.24. Fr., 20.12.23).—See B.P. 226,492; B., 1925, 703.

[Sulphide vat] dye intermediates. F. BALLAUF, F. MUTH, and A. SCHMELZER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,675,478, 3.7.28. Appl., 16.1.25. Ger., 24.1.24).—See B.P. 243,557; B., 1926, 121.

Aqueous dispersions (B.P. 296,685).—See XIV.

IV.—DYESTUFFS.

See also A., Oct., 1137, Shibuo (KOMATSU and MATSUNAMI).

PATENTS.

Sulphur dye. L. M. SHAFER, Assr. to TOWER MANUF. Co., Inc. (U.S.P. 1,675,430, 3.7.28. Appl., 18.1.26).—A mixture of *m*-tolylenediamine, dehydrothio-*p*-toluidine, and *p*-phenylenediamine (or *p*-nitroaniline) is heated with sulphur at 215—240° to give brown to greenish-bronze sulphide dyes. C. HOLLINS.

Manufacture of a yellow azo dye [for acetate silk]. E. FISCHER and C. E. MÜLLER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,684,762, 18.9.28. Appl., 20.4.27. Ger., 3.5.26).—See B.P. 270,352; B., 1928, 8.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Uses of esterified cotton. G. E. L. HIND (J. Soc. Dyers and Col., 1928, 44, 280—281).—Esterified cotton (cf. B.P. 233,701; B., 1925, 956) in lustred and non-lustred types known as "Crestol" and "Cotopa," respectively, is now commercially available. Esterification occurs throughout the entire cotton fibre and results in considerable swelling and a 20% increase in weight. Esterified cotton resists direct dyes but may be dyed with Artisil dyes (disperse dyes similar to those of the S.R.A. series), so that materials consisting of cotton and esterified cotton may be dyed in two colours by one-bath methods. Crestol has a higher tensile strength than cellulose acetate silk and gains (cellulose acetate silk loses) strength when wetted; it is unaffected by storage at high temperatures (60—70°), whereas cellulose acetate silk acquires a yellowish-brown colour and a harsh handle within a few weeks under similar conditions. Crestol is not affected by hot ironing and does not lose its lustre when boiled in neutral or weak acid solutions; it can be dry-cleaned by the usual solvents. Esterified cotton may be scoured, de-sized, bleached, and mercerised. A. J. HALL.

Esterification of cellulose and cellulose esters.
I. Velocity of nitration of cotton fibre. II. Decrease in viscosity of cellulose nitrate with duration of nitration. K. ATSUKI and M. ISHIWARA (Proc. Imp. Acad. Tokyo, 1928, 4, 382—385, 386—388).—I. The nitration of cellulose fibre is governed firstly by the rate of diffusion of the mixed acid and secondly by the rate of nitration of the successive hydroxyl groups of the cellulose molecule. The diffusion rate is given by the expression $(a-n) = ae^{-kt}$, where a = % total nitrogen in the most nitrated product, n = % total nitrogen in the product after t min., and k = constant (0.649). The rate of nitration is given by the expression $k = (1 - \sqrt{1 - m})^2/2t$, where m = the fraction nitrated in t min., and k = constant (0.0291). Diffusion to the centre of a fibre was complete in 25 min., whilst complete nitration was effected in 100 min.

II. The viscosities of cellulose nitrate solutions, in ethyl ether-alcohol mixtures or in acetone, decrease with increasing nitration time, the rate of change being given by the expression $(a-y) = ae^{-k(c-n)t}$ where a = assumed viscosity at $t = 0$, $(a-y)$ = viscosity after nitration for t min., c and n are constants, and k is a velocity coefficient. The decrease in viscosity is attributed to the depolymerisation of the molecular aggregate of cellulose nitrate during the nitration. A. E. MITCHELL.

Transverse sections of artificial silk. II. Changes in section during moistening with water. Y. KAMI and S. NAKASHIMA (J. Cellulose Inst., Tokyo, 1928, 4, 206—209; cf. B., 1927, 438).—The cross-sectional area of filaments of well-known brands of artificial silk increased by 50—60% after soaking in water, whilst the periphery alters but little. The filament tends to regain

its original form when first coagulated. Reverse changes occur during drying. A. G. POLLARD.

Bleaching of sulphite pulp. R. BERGQUIST (Papier-Fabr., 1928, 26, 593—604).—A method for the determination of p_H values of bleaching solutions is described. The effect of changes of reaction in hypochlorite bleaching baths on the nature of the bleached pulp is examined. The mechanism of bleaching is discussed on the basis that chlorination and oxidation occur simultaneously to extents which may be partially controlled through the reaction of the liquor. During bleaching there is a progressive change of reaction toward the alkaline side. Alkaline bleaching favours the production of the whitest pulp. The hypochlorite ion is the chief bleaching agent, free undissociated hypochlorous acid having little bleaching power and chlorine having not only a poor bleaching effect but a definitely injurious action on the cellulose. In alkaline bleaching liquors the hypochlorite ion is preponderant. Analysis of pulps bleached in baths of varying reaction shows that the α -cellulose content, viscosity, and copper number are affected by changes in reaction. Regions of maximum change in these values are similar, increased copper number and α -cellulose content tending to correspond with decreased viscosity and *vice versa*. Two well-defined maximum ranges occur, one on the alkaline and one on the acid side of neutrality. A. G. POLLARD.

See also A., Oct., 1079, X-Ray observations with cellulose (HERZOG and JANCKE). 1109, Sorption of vapours by fibrous or film materials (NEWSOME). 1119, Carbohydrates from pine wood (HÄGGLUND and others). Pectin and hemicellulose of flax (HENDERSON). Soluble pinewood lignin (FRIEDRICH). 1162, Cell-wall substances of plants (CANDLIN and SCHRYVER). Cell-wall of wood (RITTER).

PATENTS.

Treatment of plant materials in the preparation of fibres. Treatment of fibrous plant tissues. ABBEY SYNDE, LTD., and D. R. NANJI (B.P. 296,333—4, 29.4.27).—(A) Separation of the individual component fibres of unretted flax, grasses, etc. is effected by heating with a 0.1—0.5% solution of ammonium sulphate at 1—2 atm. for 60—20 min. to render soluble the gums and pectins. After removal of the slimy matter from the fibres by pressure, the depectinised tissue is heated with a 2—4% solution of caustic soda at 6—10 atm. for 20—60 min. and finally bleached. (B) A lustrous fibre is produced from hemp or other similar fibrous material by heating with 1—4% caustic soda solution at 10 atm. for 20—60 min., the material being bleached before or after the treatment. F. R. ENNOS.

Production of fibrous thread [from sugar cane]. J. J. DE LA ROZA, Assr. to BAGASSE PRODUCTS CORP. (U.S.P. 1,681,223, 21.8.28. Appl., 22.3.27).—Sugar cane is allowed to ferment so as to generate acids, and the fibrous part of the cane is retted with running water. H. ROYAL-DAWSON.

Washing of textile fabrics. HENKEL & CO. G.M.B.H. (B.P. 276,338, 16.8.27. Ger., 17.8.26).—A self-acting detergent effect may be obtained in washing liquors at temperatures below 70° by adding thereto urea and a

urease preparation to promote enzymic decomposition of the urea into ammonia and carbon dioxide.

D. J. NORMAN.

Protection of wool, skin materials, textiles, etc. against injurious insects. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 295,742, 18.5.27).—The material is treated with, e.g., 10 times its weight of 0.2% hot or cold aqueous solution of a compound of the type $X(HF)_n$, where X is an organic or alkali salt free from fluorine, or a metal fluoride. Suitable compounds include tripotassium difluorodisulphate, monopotassium monofluorophosphate, and the additive product sodium *p*-toluenesulphonate + 2HF. Organic or inorganic acids or salts may be added to the bath, if desired. (Cf. B.P. 173,536; P., 1922, 138A.) D. J. NORMAN.

Recovery of cellulose from plants such as beechwood, bamboo, etc. R. RUNKE (B.P. 296,547, 24.8.27).—The comminuted raw material is steeped in water or solutions of acid or basic substances, e.g., tin chloride or caustic soda, either at ordinary or elevated temperatures for a suitable time. After removal from the liquid and reduction to the state of fine fibres, the mass impregnated with alkali is chlorinated while suspended in water, centrifuged, and treated with a 0.5–1% solution of caustic soda, the oxidation and chlorination process being repeated until a pure white cellulose is obtained.

F. R. ENNOS.

Manufacture of cellulose acetate. G. W. MORDEN (B.P. 294,415, 25.11.27).—Cellulose is treated with sulphuryl chloride, acetic acid, and acetic anhydride, and acetylation is subsequently completed at below 25° in a mixture of acetic acid, acetic anhydride, and sulphuric acid. Hydrolysis to an acetone-soluble cellulose acetate is carried out in the usual way.

D. J. NORMAN.

Manufacture of cellulose acetate. E. S. FARROW, JUN., Assr. to EASTMAN KODAK CO. (U.S.P. 1,679,966, 7.8.28. Appl., 5.9.25).—Cellulose is treated with concentrated hydrochloric acid, free from oxidants, at 15° for 2–6 hrs. before acetylation. C. HOLLINS.

Production of cellulose ester and ether compositions. H. J. HANDS, and SPICERS, LTD. (B.P. 294,008, 27.8.27).—Mixtures of diphenyl monocresyl phosphate and phenyl dicresyl phosphate are used as plasticisers for cellulose ester and ether compositions, the relative proportions of these two compounds being so chosen that the final mixture corresponds, as regards its content of phenyl, cresyl, and phosphoric anhydride, to a mixture containing 45–65% (preferably 55%) of triphenyl phosphate and 55–35% (preferably 45%) of tricresyl phosphate. In this way the reduction of inflammability imparted by triphenyl phosphate and the pliability imparted by tricresyl phosphate may be obtained, without the disadvantages arising from the tendency of the former to crystallise out and the latter to give a greasy character to the film. D. J. NORMAN.

Cellulose ester composition. R. L. KRAMER, Assr. to E. I. DU PONT DE NEMOURS & CO. (U.S.P. 1,676,612, 10.7.28. Appl., 6.9.24).—As softeners for cellulose nitrate especially in the manufacture of artificial leather there are claimed β -aryloxyethyl phthalates or

laurates. β -Phenoxyethyl phthalate, β -o-(*m*- or *p*-)toloxyethyl phthalate, and β -o-(*m*- or *p*-)toloxyethyl laurate, m.p. -1° , are described. C. HOLLINS.

Reducing the viscosity of cellulose ethers and products thereof. C. U. PRACHEL and L. E. BRANCHEN, Assrs. to EASTMAN KODAK CO. (U.S.P. 1,679,943, 7.8.28. Appl., 21.12.25).—A cellulose ether is treated with sludge acid until the viscosity is reduced by at least one third.

C. HOLLINS.

Production of foils, films, bands, etc. from viscose and similar cellulose solutions. WOLFF & CO., E. CZAPEK, and R. WEINGAND (B.P. 277,309, 12.8.27. Ger., 13.9.26).—In the manufacture of films by extruding viscose solution on to, e.g., a rotating drum half immersed in a coagulating bath, there occurs a small interval of time during which the surface of the drum covered by the coagulated film becomes quite dry. If, therefore, the film is removed during this interval the surface of the drum remains clean and free from adhering fragments of viscose. The factors influencing the time which elapses between the application of the film to the drum and the point at which the surface of the drum becomes dry are adjusted so that the required time interval occurs when, e.g., the drum has made about three quarters of a revolution starting from the point of application of the viscose solution.

D. J. NORMAN.

Production of cellulose membranes, bands, and films. O. SINDL (B.P. 284,725, 4.2.28. Ger., 5.2.27).—Immediately below the slit nozzle through which the viscose solution flows is placed a second slit nozzle through which the coagulant is delivered down an inclined surface into the coagulating bath. In this way the viscose film floats down to the bath on a thin film of coagulant, whereby the underside of the film becomes coagulated while the upper part is still sufficiently fluid to permit the gases evolved to escape freely.

D. J. NORMAN.

Manufacture of artificial materials from viscose. I. LILIENFELD (B.P. 264,161, 5.1.27. Austr., 5.1.26).—Viscose silk of high strength both in the wet and dry state is obtained if, after coagulation in an acid bath of the ordinary type, the filaments are led into a second bath containing one or more strong mineral acids, e.g., sulphuric acid of 50–85% strength (preferably at least 65%), optionally containing suitable organic or inorganic substances such as glucose, sodium bisulphate, etc. The temperature of the strong acid bath may be between -10° and 40° , and the length of travel of the filaments may be 3–60 cm. or up to 2 m. The strength of the filaments is improved if additional stretch is applied to them in one or other of the baths or during their travel to the collecting device. Unmatured or but slightly matured viscose in the preparation of which 75–150% of carbon disulphide on the weight of cellulose has been used is particularly suitable.

D. J. NORMAN.

Manufacture of artificial filaments, threads, etc. COURTAULDS, LTD., H. J. HEGAN, and E. HAZELEY (B.P. 294,279, 21.4.27).—The annular cake of artificial filaments produced by box-spinning processes is more easily washed (or otherwise treated) centrifugally if a layer of pervious cloth, a wire gauze, or similar device

is interposed between the cake and the bottom of the box, so that the treating liquor can pass between them.

D. J. NORMAN.

Treatment of fabrics or articles containing fibres or threads of organic derivatives of cellulose. BRIT. CELANESE, LTD. (B.P. 274,074, 1.7.27. U.S., 8.7.26).—Ordinary carbonisation processes can be applied for the removal of vegetable fibres (including regenerated cellulose silks) from mixed fabrics containing them in association with fibres derived from cellulose esters or ethers without impairing the dyeing or dye-resisting properties of the cellulose derivative or seriously reducing its lustre. Delustreing may, moreover, be entirely prevented by adding sodium sulphate to the carbonising bath, in quantity depending on the duration of treatment.

D. J. NORMAN.

Supports made of cellulose derivatives for use in the manufacture of sheets of material soluble in organic solvents. I. G. FARBENIND. A.-G. (B.P. 279,047, 8.9.27. Ger., 16.10.26).—The support is made of a layer of a cellulose derivative the surface of which has been so treated, e.g., hydrolysed, that it consists wholly or partly of regenerated cellulose, and is therefore unaffected by organic solvents.

D. J. NORMAN.

Manufacture of cardboard. G. E. HEYL, and HYCOLITE LIQUID WALLPAPER MANUF. CO., LTD. (B.P. 294,442, 26.1.28).—Fibrous pulp or waste paper is finely comminuted in a dry condition in a high-speed disintegrator, and the resulting powder is mixed with 30–50% of water and 4–8% of a binder such as dextrin. This paste is then beaten in a colloid mill and formed into sheets.

D. J. NORMAN.

Manufacture of cellulose derivatives. L. LILIENFELD (U.S.P. 1,680,224, 7.8.28. Appl., 22.5.26. Austr., 30.5.25).—See B.P. 252,654; B., 1926, 1009.

Cellulose ether and process of making same. L. LILIENFELD (U.S.P. 1,683,831, 11.9.28. Appl., 16.6.23. Austr., 13.7.22).—See B.P. 200,834; B., 1925, 202.

Preparation of cellulose ethers and alkali cellulose. L. LILIENFELD (U.S.P. 1,683,681, 11.9.28. Appl., 16.6.23. Austr., 13.7.22).—See B.P. 200,827; B., 1925, 67.

Preparation of alkali cellulose and cellulose ethers. L. LILIENFELD (U.S.P. 1,683,682, 11.9.28. Appl., 16.6.23. Austr., 13.7.22. Renewed 5.4.27.).—See B.P. 200,816; B., 1925, 67.

Apparatus [measuring pumps] for use in the manufacture of artificial silk or other operations in which liquids are supplied under pressure. BRIT. CELANESE, LTD., and E. KINSELLA (B.P. 293,325, 23.12.26 and 22.10.27).

[Twisting and winding threads in] manufacture of artificial silk or like threads and apparatus therefor. BRIT. CELANESE, LTD., and S. A. WELCH (B.P. 293,371, 4.4.27).

Cellulose waste liquors (B.P. 269,909).—See VII. **Treatment of cellulose films** (B.P. 281,663).—See XI. **Mixed lacquers etc.** (B.P. 296,796).—See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Influence of anions of mordants on the shade of Alizarin Red lake. P. P. VICTOROV (Rev. gén. Mat. col., 1928, 32, 253–260).—An Alizarin Red printing paste to which iron salts have been added gives the best results in presence of a reducing agent (sodium hyposulphite) and the worst if an oxidant (potassium chlorate) is present. This is due to the solubility of the ferrous lake and the non-formation of the insoluble ferric lake in presence of reducing agents. Addition of thiocyanate protects the lake from iron contamination, but is ineffective when iron salts are added. For brightness and depth of shade a slow hydrolysis of the mordant salt is desirable: mordant salts of acids which are volatilised during steaming are too rapidly hydrolysed. Mordant formates, however, in spite of this give good results on account of the reducing power of formic acid. The influence of the anion of mordant salts is thus complicated by the reducing effect and the rate of hydrolysis.

C. HOLLINS.

Production of Thioindigo Scarlet on the fibre. M. BAUMANN (Sealed Note No. 1955, 24.11.09. Bull. Soc. Ind. Mulhouse, 1928, 94, 372–373). Report by BATTEGAY and J. SCHMID (*Ibid.*, 374).—Cotton and other fabrics may be dyed with Thioindigo Scarlet by padding with an alkaline solution containing phenylthioglycine-*o*-carboxylic acid and isatin bisulphite and steaming, whereby condensation and oxidation of these intermediates occurs. Phenylthioglycine-*o*-carboxylic acid is preferred to thioindoxyl since it is soluble in borax, whereas the last-named substance requires sodium carbonate for its dissolution, and premature formation of Thioindigo Scarlet occurs in the padding liquor when sodium carbonate is present; the bisulphite compound of isatin is used instead of isatin for the same reason. Fabric may be dyed satisfactorily in pale pink to deep scarlet shades by padding with a liquor containing 10 g. of phenylthioglycine-*o*-carboxylic acid, 30 g. of borax, 2.8 g. of sodium carbonate, 10 g. of isatin, and 16 g. of sodium bisulphite per litre, then drying (a pink colour is thereby developed), steaming for about 5 min., and washing in a hot 1% solution of sodium carbonate for completion of the condensation. White and coloured reserves may be obtained by means of resist pastes containing a stable form of sodium hyposulphite, e.g., Rongalite. Battagay and Schmid draw attention to similar processes described in G.P. 184,956 of 1905 and 229,369 of 1909 (cf. F.P. 407,226; B., 1910, 482).

A. J. HALL.

Action of light on cotton dyed with vat dyes. F. SCHOLEFIELD and C. K. PATEL (J. Soc. Dyers and Col., 1928, 44, 268–274).—Cotton and viscose silk materials dyed with one of a large number of yellow and orange vat dyes (Indanthrene Yellow G and Alizaranthrene Yellow 6R excepted) and exposed to light and air immediately after dyeing and while the vat dye is in its leuco-condition, become tendered by oxidation; the dye itself may also be simultaneously oxidised with consequent partial or complete decolorisation, or if a second dye is present (especially Ciba Blue 2B) this may be oxidised in preference to the cotton or first dye. These effects are also produced when the dyed fabric is oxidised in the dark before exposure to light (sunlight

or light from a Fadeometer), but are not produced if again washed before exposure; tendering occurs if the washed fabric is further impregnated with a dilute solution of caustic soda containing sodium hyposulphite or hydrogen peroxide and then exposed to light and air. By means of the starch-iodide test it was shown that an active oxidising agent (probably hydrogen peroxide) was formed during the oxidation of cotton dyed with Cibacron Yellow R. Certain direct dyes, including Diamine Fast Yellow and Chlorazol Fast Orange R, behave similarly to vat dyes in promoting tendering. The tendering of cotton when so exposed is rapid and may be serious in large-scale dyeing. *E.g.*, cotton fabric lifted successively 5 times (10 min. exposure each time) from the dye liquor and exposed to light and air while being dyed with 20% Cibacron Orange R paste had copper numbers of 0.2 (original fabric), 0.3 (dyed fabric not exposed), 0.7, 1.3, 1.5, 1.7, and 1.75 (after five successive exposures). Viscose silk is particularly susceptible to tendering if exposed during dyeing with Cibacron Orange R. The destructive action of sunlight is not appreciably affected by transmission through plate glass $\frac{1}{4}$ in. thick or didymium glass opaque to yellow light.

A. J. HALL.

Azoic [Naphthol AS] colours on wool. S. C. TURNER (J. Soc. Dyers and Col., 1928, 44, 276—280; cf. Rath, B., 1928, 638).—Naphthols of the Naphthol AS series are substantive to wool and may be applied without deleterious effect to this fibre from solutions the temperature and alkalinity of which do not exceed 50° and 1.25 g./litre of caustic soda, respectively. The presence of sodium chloride in the naphthol solution diminishes the harmful effect of the caustic soda on wool and increases the absorption of the naphthol by wool, but it tends to salt out the naphthol, particularly with Naphthols AS-SW, AS-BR, and AS-BS, though less with Naphthols AS-G, AS-RL, and AS-D; it is suitable to use 60—80% (on the wool) of sodium chloride. The presence of sodium chloride in the naphthol solution particularly assists the absorption by wool of Naphthols AS-BR, AS-TR, and AS-G. Leonil S can replace soluble oils used in preparing Naphthol AS solutions and allows a considerable reduction in the amount of caustic soda used; it diminishes the susceptibility of such solutions to salting-out. Wool exerts its maximum affinity for Naphthols AS-SW, AS-BO, AS-G, and AS-BS at 30°; for Naphthols AS-D, AS, AS-BG, and AS-RL at 40°; and for Naphthols AS-TR, AS-BR, and AS-OL at above 50°; the tone of the resulting dyeings changes with a rise of temperature in the case of the three last-named naphthols. The rate of absorption of naphthols by wool is very rapid during the first 20 min.; the most satisfactory durations for naphtholating for deep and pale shades are 30 and 45 min., respectively. Naphtholated wool can be partially stripped by soaping, but not by washing with water. Diazo compounds stain wool brown during development unless a 1% excess of acetic acid is added to the diazo solution; the stain on damaged fibres is proportional to the degree of damage. It is essential that naphtholated wool should be completely developed by the diazo compound, since naphthols on wool readily become brown by exposure to light. The

fastness to rubbing and light of azoic dyes on wool is respectively greater than and equal to that on cotton.

A. J. HALL.

Novel [colour] effects on half-silk materials. P. DOSNE (Sealed Note No. 1386, 4.4.03. Bull. Soc. Ind. Mulhouse, 1928, 94, 358). Report by L. PAULUS (*Ibid.*, 358—359).—Coloured textiles such as imitation "homespun" are obtained from yarns manufactured by spinning together cotton and waste silk and then dyeing with dyes (*e.g.*, sulphur dyes) having an affinity for only one type of fibre.

A. J. HALL.

Dyeing of [cellulose] acetate silk. V. KARTASCHOV and G. FARINE (Helv. Chim. Acta, 1928, 11, 813—836; cf. B., 1926, 49, 188).—A detailed study has been made of the dyeing of cellulose acetate silk (Celanese) by 15 water-insoluble anthraquinone derivatives. Determinations of the solubilities of these compounds in absolute alcohol show that the most soluble derivatives contain either a nuclear methyl group or a methylamino-group. The diaminoanthraquinones are more soluble than either the mono- or tetra-amino-derivatives, whilst the introduction of a hydroxyl group causes a slight diminution in the solubility. The solubilities of the dyes in the Celanese were determined by treating the material with an aqueous suspension of the dye at 60°, washing out the superficial colouring matter, and dissolving the dyed silk in acetone. During this process superficial crystallisation of the dyestuff on the fibre takes place followed by dissolution. The solubilities are independent of the concentration of the suspension and the size of the particles, and are analogous to those in alcohol. Determinations of the partition ratios of the dyestuff, silk, and alcohol show that for varying amounts of silk the values are the same for each dyestuff. These results indicate that the molecular changes in the colouring matters (association or polymerisation) are the same in both solvents, and also that adsorption does not occur.

II. BURTON.

Coloured reserves under sulphur dyes [by printing]. L. GOUBYRIN (Sealed Note No. 1933, 28.9.09. Bull. Soc. Ind. Mulhouse, 1928, 94, 367—370; cf. B., 1928, 479). Report by V. SCHWARTZ (*Ibid.*, 370—371).—Basic and insoluble azo coloured reserves under sulphur colours may be obtained simultaneously by reason of the fact that, during steaming, the formaldehyde compound of β -naphthol reacts with resorcinol in the presence of zinc chloride thereby yielding free β -naphthol capable of coupling with a diazo compound and a condensation product of resorcinol and formaldehyde capable of fixing basic dyes satisfactorily. Schwartz reports favourably on the process, the basic dyes fixed on the naphthol-formaldehyde mordant being fast to washing.

A. J. HALL.

Esterified fibre. VICTOROV. **Bleaching sulphite pulp.** BERGQUIST.—See V.

PATENTS.

Preparation of effect threads. CHRM. FABR. VORM. SANDOZ (B.P. 280,493, 12.5.27. Ger., 13.11.26).—Effect threads resistant to substantive dyes may be obtained by acetylating cellulose in any form in a

mixture the activity of which has been so moderated, by adjustment of the quantity of catalyst and the temperature, that the cellulose is converted direct into mono- and di-acetates whilst retaining the original form, strength, and elasticity of the original material. A suitable acetylating mixture per kg. of cotton contains 3 kg. of acetic anhydride, 5 kg. of glacial acetic acid, and 2.5 g. of sulphuric acid. The reaction is allowed to proceed at 15°, and after two days a dye test is made to ascertain if acetylation has proceeded sufficiently far to render the fibre completely resistant to substantive dyes. D. J. NORMAN.

Phosphate reducer for bottom chrome dyeing. N. NEVILLE, ASST. to FEDERAL PHOSPHORUS Co. (U.S.P. 1,675,459, 3.7.28. Appl., 29.10.24).—Sodium hydrogen pyrophosphate, with or without sodium hydrogen sulphate, is added to the dichromate bath in place of lactic acid or other assistant. Brighter dyeings are claimed. C. HOLLINS.

Process and apparatus [with submerged burner] for dyeing or washing articles of clothing, fabrics, etc. C. F. HAMMOND and W. SHACKLETON (B.P. 296,525, 13.4.27).

Colouring of rubber etc. (B.P. 296,461).—See XIV.
Dyeing of leather (F.P. 618,667).—See XV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Wet process for working-up of phosphorites. S. I. WOLFKOVICH and V. P. KAMZOLKIN (J. Chem. Ind., Moscow, 1928, 5, 474—480).—Phosphorite meal is treated with a mixture of sulphuric acid and ammonium sulphate, and the residue, containing 45—70% of calcium sulphate, is treated with ammonia and carbon dioxide, to form ammonium sulphate and calcium carbonate. The latter again is treated with oxides of nitrogen to yield calcium nitrate. No waste products are formed, both the ammonium phosphate and the calcium nitrate obtained being useful final products, whilst no basic material is lost. R. TRUSZKOWSKI.

Reduction of barium carbonate by aluminium. E. F. LINNORST and H. SCHLUNDT (Amer. Electrochem. Soc., Sept., 1928. Advance copy. 4 pp.).—On heating a finely-divided mixture of barium carbonate and aluminium in the molar proportion of 1 : 2 to about 400° a vigorous reaction occurs and the temperature rises to about 1400°. The product is a hard grey mass consisting of barium aluminate with small amounts of barium and aluminium carbides. With a much smaller proportion of aluminium the reaction was incomplete, and with coarser-grained aluminium powder no reduction occurred even when the mixture was heated to relatively high temperatures. H. J. T. ELLINGHAM.

Electrometric titration of hypochlorite and hypochlorite-carbonate mixtures. A. RIUS and V. ARNAL (Amer. Electrochem. Soc., Sept., 1928. Advance copy. 8 pp.).—Electrometric titration of chlorine water with 0.1N-sodium hydroxide gives a point of inflexion on the titration curve corresponding with the formation of sodium chloride and hypochlorous acid, and another point of inflexion when the latter is

converted into sodium hypochlorite. The platinum electrode which is immersed in the solution acts as a chlorine electrode. In a similar titration of a solution of sodium hypochlorite and carbonate with a 0.1N-hydrogen peroxide, 0.1N-sulphuric acid solution points of inflexion were obtained corresponding with (a) neutralisation of free sodium hydroxide and half of the carbonate and (b) neutralisation of all the carbonate, whilst there is a sharp fall in potential when the hypochlorite is completely converted into chloride. Better values for hydroxide and carbonate were obtained by titrating with a solution containing a larger proportion of hydrogen peroxide so that the hypochlorite is decomposed while the solution is still alkaline and the sharp fall in potential thus precedes the points of inflexion. H. J. T. ELLINGHAM.

Determination of small quantities of carbon monoxide and methane in mixtures of nitrogen and hydrogen. K. KELLER and W. KLEMP (Ber. Ges. Kohlentechn., 1927, 2, 246—249; Chem. Zentr., 1927, II, 2253—2254).—The gas mixture is passed successively through a porcelain tube containing copper oxide at 500—520° whereby hydrogen and carbon monoxide are oxidised, through carbon dioxide absorption apparatus containing barium hydroxide solution (1 c.c. \equiv 1 c.c. CO₂ at 20° and 760 mm.), through a copper oxide tube at 850° to oxidise the methane, through a second carbon dioxide absorption apparatus, and into a burette in which the nitrogen is collected and measured over water. L. A. COLES.

Recovery of bromine from iron bromide. P. HÖFER (Kali, 1927, 21, 222—224; Chem. Zentr., 1927, II, 2033).—95% of the bromine in commercial iron bromide, Fe₃Br₈, can be recovered by heating in a current of air. The ferric bromide is decomposed at 180° into bromine and iron oxide, and similar decomposition of the ferrous bromide begins at 310°. About 3.1% of hydrogen bromide is also formed. L. A. COLES.

See also A., Oct., 1090, **Silver hydrosols** (VOIGT).
Colloidal gold solutions (VON WEIMARN). 1095, **Decomposition of alkaline-earth sulphates** (ZAWADSKI and others). **Dehydration of hydrated salts** (RAKUZIN). 1099, **Decomposition of nitrogen pentoxide** (SPRENGER). **Kinetics of nitrous acid** (ABEL and others). 1101, **Electrolytic preparation of cuprous oxide** (ABEL and REDLICH). 1103, **Effect of alkalinity on basic cupric sulphates** (NELSON). **Basic magnesium carbonate** (NISHIMURA). **Calcium nitride** (DUTOIT and SCHNORF). 1104, **Hydrated tri-calcium aluminate** (TRAVERS and SEHNOUTKA). **Denigès' phospho-ceruleomolybdenum compound** (VERDA). **Uranyl sulphate** (COLANI). 1105, **Analysis by sedimentation** (ARRHENIUS and RIEHM). 1106, **Detection of sulphide and thiosulphate** (FEIGL). 1107, **Microdetermination of sulphuric acid** (YAMAZAKI). **Determination of persulphate** (SCHWICKER). **Detection of phosphate in presence of arsenate and molybdate** (FEIGL and KRUMHOLZ). **Indophenol reaction in inorganic chemistry** (STIEPOVICH and SA). 1108, **Determination of salts in solution** (BOU-TARIC and PERREAU). **Micro-determination of calcium** (ROGOZINSKI). **Detection of magnesium in**

rocks (FEIGL). Colour reactions for magnesium (BARNES). Colorimetric determination of iron (VAN DER VLUGT). 1009, Photo-electric spectrophotometric analysis (VON HALBAN and ZIMPELMANN). Potentiometric determination of iron, copper, and arsenic (ZINTL and SCHLOFFER). Determination of bismuth (PINKUS and DERNIES).

Alloys etc. for nitric acid industry. WAESER.—See X. Hydrogen generator. GARDINER and HULETT.—See XI. Saltpetre and sodium nitrite as pickling agents. RIESS and others.—See XIX. Chlorine water. MIKÓ.—See XX.

PATENTS.

Manufacture of nitric acid. W. R. ORMANDY (B.P. 296,121, 25.5.27).—Oil or other fuel is burnt in a flame submerged in water in presence of a catalyst consisting of a compound of a metal capable of forming a number of oxides, *e.g.*, nickel, vanadium, chromium, etc. Methods of introducing the catalyst into the oil, the flame, or the combustion air are described.

W. G. CAREY.

Production of phosphoric acid. H. MEHNER (Swiss P. 120,510, 21.7.25).—The mixture of phosphorus and carbon monoxide obtained by heating phosphates with coke and silica is burnt immediately it leaves the reaction zone, and phosphorus pentoxide is recovered from the products of combustion.

L. A. COLES.

Manufacture of hydrogen cyanide from formamide. P. LA F. MAGILL and P. J. CARLISLE, ASSRS. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,675,366, 3.7.28. Appl., 14.1.27).—Formamide vapour is passed rapidly through brass tubes (diam. not more than 1 in.) immersed in a bath at 450–650°.

C. HOLLINS.

Manufacture of materials containing iron oxide and of paint therefrom. F. RIVERS (B.P. 296,598, 17.11.27).—A product containing iron oxide, silica, and magnesia is made from clay containing at least 25% of iron oxide, the clay being balled into lumps, burned for 12 hrs., crushed, and screened. The material is then incorporated with linseed oil and turpentine to form paint.

W. G. CAREY.

Production of alumina. M. BUCHNER (B.P. 283,117, 3.1.28. Ger., 3.1.27).—Aluminiferous material, *e.g.*, clay, is heated for several hours at 80–100° with a deficiency of nitric acid to form basic aluminium nitrate, the solution is concentrated by partial evaporation, cooled to 0° to crystallise the basic nitrate, and the product heated *in vacuo* with water or steam. W. G. CAREY.

[Continuous] production of red lead. E. HAYWARD (B.P. 295,975, 22.2.27).—Granular or finely-divided lead, lead carbonate, or massicot is heated at 450–500° and under 20–30 atm. in a reaction chamber provided with intermediate chambers and automatic valves for introducing and discharging material without loss of air pressure, and with means for conveying the material through the chamber; air and/or oxygen is circulated under pressure through the chamber in counter-current to the material. The air-oxygen mixture after use is re-circulated with the addition of fresh air or oxygen.

W. G. CAREY.

Production of sodium sulphide from sodium hydrosulphide. P. KIRCHEISEN (G.P. 449,282, 25.12.26).—Sodium hydrosulphide solution is treated with carbon dioxide until it contains 1 mol. of sodium carbonate per 2 mols. of sodium hydrosulphide, and is then treated with lime to yield calcium carbonate and sodium sulphide.

L. A. COLES.

Production of porous sodium sulphide readily soluble in water. B. ROOS & CO. (G.P. 449,284, 1.8.26).—The reduction process is effected under such conditions that the product is as pure as possible, and, before the reduction is finished, the melt is transferred from the furnace to another vessel in which the reduction is completed.

L. A. COLES.

Production of sodium sulphide. I. G. FARBERNIND. A.-G., Assees. of A. SCHÄFER (G.P. 449,584, 19.1.26).—Sodium sulphate is reduced to the sulphide by heating with coke in an air-tight furnace provided with a flat, rotating hearth, the materials being charged in continuously through a water-cooled device. L. A. COLES.

Production of technically pure alkali hydrosulphides. B. REINHARDT (G.P. 449,283, 22.5.24).—The solution obtained by treating barium hydrosulphide with an alkali chloride and subsequently removing precipitated barium chloride is treated with carbon dioxide or carbonates to precipitate residual barium salts.

L. A. COLES.

Manufacture of solid sodium hydrogen sulphite or sodium sulphite. CHEM. FABR. KALK GES.M.B.H., and H. OEHME (G.P. 446,525, 5.4.25. Addn. to G.P. 440,380; B., 1927, 777).—Sodium hydrogen sulphite solution is neutralised with sodium hydrogen carbonate, and the resulting carbon dioxide is absorbed in sodium carbonate solution. The neutral sulphite solution or suspension is again saturated with sulphur dioxide and the process repeated as often as is necessary to obtain a solid sulphite.

A. R. POWELL.

Treating residual liquors accruing from the treatment of cellulose material [using sodium sulphite]. P. A. BARBOU and R. DELVILLE [Soc. BARBOU & CIE.] (B.P. 269,909, 21.4.27. Fr., 21.4.26).—The waste liquor is agitated with milk of lime at 75° whereupon calcium sulphite is precipitated, leaving a supernatant solution containing caustic soda and a sodium organic salt. The calcium sulphite is separated by decantation and converted into calcium bisulphite by treatment with sulphur dioxide, whilst the decanted liquor is agitated with calcium bisulphite to give an insoluble calcium organo-complex and a mixture of sodium sulphite and bisulphite. After settling, the supernatant liquor is drawn off and treated with sufficient caustic soda to form normal sodium sulphite for use in the treatment of further quantities of cellulosic material. The calcium organo-complex is dry-distilled to recover acetic acid, acetone, etc. D. J. NORMAN.

Utilisation of [production of sodium thiosulphate and carbon disulphide from] trithiocarbonate solutions. SILESIA VER. CHEM. FABR., Assees. of H. KLEIN (G.P. 449,604, 13.10.26).—Solutions containing trithiocarbonates, obtained in the preparation of aromatic substituted thiocarbamides, are treated with sulphur

dioxide or a solution of it in concentrated sodium thiosulphate solution. L. A. COLES.

Purification of sodium stannate solution. C. L. READ and B. M. O'HARRA, Assrs. to AMER. SMELTING & REFINING Co. (U.S.P. 1,681,497, 21.8.28. Appl., 3.7.26).—Lead and copper are precipitated by adding sodium sulphide and are removed; antimony, selenium, and tellurium are then precipitated by heating with tin in an oxidisable condition. F. G. CLARKE.

Production of ammonium nitrate. ELEKTRIZITÄTSWERK LONZA (F.P. 623,265, 18.10.26. Switz., 23.10.25 and 17.6.26).—Crystalline ammonium nitrate prepared as described in Swiss P. 118,962 (B., 1928, 568) is purified by washing with concentrated ammonium nitrate solution, or manganese is precipitated as its hydroxide before crystallisation. Small quantities of manganese remaining in the mother-liquor are precipitated by intimate treatment with air.

L. A. COLES.

Production of ammonium phosphate. S. G. S. DICKER. From CHEM. PRODUCTS Co. (B.P. 297,009, 10.6.27).—Phosphatic rock etc. is treated with ammonium bisulphate solution and the solution, after removal of calcium sulphate, is treated with ammonia to form triammonium phosphate, which is crystallised out, removed, and heated to yield a mixture of mono- and diammonium phosphates with liberation of ammonia. The calcium sulphate obtained in the first stage is treated with ammonium carbonate to yield ammonium sulphate and this, together with the ammonium sulphate in the mother-liquor from the crystallisation of the triammonium phosphate, is converted into the bisulphate for re-use by heating or by electrolytic treatment.

L. A. COLES.

Manufacture of barium carbonate. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G., Assces. of K. THELEN (G.P. 446,863, 24.5.24).—Barytes is reduced to thelen sulphide by heating in an atmosphere of carbon monoxide, the product is leached with water, and the solution treated with the carbon dioxide formed in the reduction process.

A. R. POWELL.

Conversion of barium carbonate into barium oxide. P. ASKENASY and R. ROSE (G.P. 444,122, 6.3.25. Addn. to G.P. 443,237; B., 1928, 51).—Barium carbonate is heated with an excess of carbon and the excess is removed by passing steam over the hot mass.

A. R. POWELL.

Treatment of barium and strontium sulphates. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G., Assces. of F. ROTHE and H. BRENEK (G.P. 444,861, 14.11.25. Addn. to G.P. 443,320; B., 1928, 51).—Before passing the reducing gases over the glowing sulphate mass they are freed from difficultly combustible constituents by passing through a purification chamber.

A. R. POWELL.

Preparation of a finely-powdered carbide from calcium carbide. H. WITTEK (G.P. 446,410, 15.5.26).—The stream of molten calcium carbide from an electric furnace is atomised by means of a blast of nitrogen in the upper part of a water-cooled shaft. For the preparation of cyanamide cooling is so regulated that the temperature of the mass is brought down only to that at

which absorption of nitrogen is rapid, and the gas stream is charged with particles of a granulated material which does not retard the reaction.

A. R. POWELL.

Manufacture of calcium nitrate together with alumina and phosphorus. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,832, 16.6.27).—Raw phosphate is fused with coal and sufficient aluminous material to produce an easily friable slag, and after recovery of the phosphorus the slag is treated with nitric acid, the silica collected on a filter, and the alumina precipitated with calcium carbonate or milk of lime with the addition of an oil, e.g., coal-tar oil or tetrahydronaphthalene, to lower the surface tension and ensure rapid filtration of the calcium nitrate solution.

W. G. CAREY.

Preparation of metal bromides. J. H. VAN DER MEULEN (B.P. 285,915, 23.2.28. Ger., 25.2.27).—The formation of bromates and hypobromites is prevented by treating a non-acid compound, e.g., oxide, carbonate, of the desired metal with bromine in the presence of water and a reducing agent which, during the process, is converted into water and/or gases. Such agents may be organic or inorganic compounds containing nitrogen, e.g., urea, cyanamide, ammonium cyanide, etc. When nitrides, formates, oxalates, etc. are used, the salts should be those of the appropriate metal.

W. G. CAREY.

Manufacture of bleach liquor as by-product in salt purification. V. YNGVE (U.S.P. 1,683,431, 4.9.28. Appl., 12.1.27).—The hydroxide sludge obtained by treatment of the brine with caustic soda (cf. U.S.P. 1,520,920; B., 1925, 131) is separated, and chlorinated until the magnesium hydroxide is on the point of being attacked. The resulting calcium hypochlorite bleach liquor is separated from the remaining sludge.

F. G. CLARKE.

Production of magnesium phosphate. VER. F. CHEM. & MET. PROD. (G.P. 449,288, 4.8.25. Addn. to G.P. 447,393; B., 1928, 603).—Mixtures of magnesium chloride and calcium phosphate are heated in the presence of chlorine or gases containing it.

L. A. COLES.

Production of aluminium chloride. T. R. HAGLUND (Swiss P. 120,514, 2.4.25. Swed., 17.4.24).—Aluminiferous material is fused with reducing agents and material containing sulphur to yield a product containing aluminium sulphide, and this is treated with chlorinating agents.

L. A. COLES.

Treatment of minerals containing aluminium. W. G. BJORKSTEDT (U.S.P. 1,681,921, 28.8.28. Appl., 26.1.27).—The minerals are heated with ammonium sulphate, and, after leaching, the aluminium in the solution is precipitated with ammonium sulphite.

H. ROYAL-DAWSON.

Production of pure lead chloride and potassium nitrate. SILESIA VER. CHEM. FABR., Assces. of P. SCHLÖSSER, K. BARTSCH, and G. ALASCHENSKI (G.P. 449,739, 18.11.25).—Crude material containing lead is treated with nitric acid, excess acid is neutralised and iron is precipitated by the addition of lead carbonate, lead chloride is precipitated by the addition of potassium chloride, and, after removal of final traces of lead by precipitation as the sulphate, potassium nitrate

is crystallised from the solution, the mother-liquor being returned to the cycle of operations.

L. A. COLES.

Manufacture of lead arsenate. GEBR. BORCHERS A.-G., and F. BORCHERS (G.P. 446,409, 15.6.26).—A solution of a soluble arsenate is treated with a deficiency of lead chloride and the reaction completed by addition of lead nitrate.

A. R. POWELL.

Preparation of lead tetra-alkyl. H. W. DAUDT (B.P. 283,913, 14.10.27. U.S., 20.1.27).—An alkyl halide is added to a suspension of magnesium and a lead salt in ether, or magnesium is added similarly to the other components, the temperature being maintained (preferably for about 12 hrs.) at 28–40°. After addition of water the tetra-alkyl is removed by steam distillation.

B. FULLMAN.

Production of nickel and/or cobalt carbonyl. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,558, 5.9.27).—Carbon monoxide is caused to react under pressure with porous, loose nickel and/or cobalt metal obtained by leaching the ores or intermediate metallurgical products with aqueous ammonia or solutions of ammonium salts containing free ammonia, treating the resulting complex ammonium compounds to obtain hydroxides or basic salts, and reducing them with hydrogen at a low temperature.

W. G. CAREY.

Electrolytic production of hydrogen and oxygen. ELEKTRIZITÄTS A.-G. VORM. SCHUCKERT & Co. (G.P. 449,603, 23.1.26).—Contamination of the oxygen in the decomposition apparatus is permitted to a greater extent than usual, and the production of an explosive mixture is prevented by diluting the gas with a non-combustible gas, *e.g.*, nitrogen, air, or oxygen, supplied through an injector for removing the gas from the apparatus in a proportion regulated automatically according to the volume of oxygen formed.

L. A. COLES.

Preparation of hydrogen. F. GÜLKER (G.P. 446,488, 31.7.26).—A mixture of water-gas with a slight excess of steam over that required to convert the carbon monoxide into dioxide is passed over a heated mixture, in granular form, of a catalyst and a substance capable of combining with the carbon dioxide produced. The catalytic mass is regenerated by heating in a current of air.

A. R. POWELL.

Production of concentrated oxides of nitrogen. I. G. FARBENIND. A.-G., Assees. of R. GRIESSBACH and O. SCHLIEPHAKE (G.P. 449,606, 17.6.26).—Gas mixtures containing oxides of nitrogen are brought in contact with liquid air, and the solidified oxides of nitrogen are recovered from the cooling agent by filtration.

L. A. COLES.

Production of phosphorus and ferrosilicon. I. G. FARBENIND. A.-G. (B.P. 290,971, 16.4.28. Ger., 21.5.27).—Ferrophosphorus is mixed with the requisite amount of silicon, ferrosilicon, or mixtures forming silicon for the production of commercial ferrosilicon, and the mixture is fused in an electric furnace, the phosphorus being recovered by condensation.

W. G. CAREY.

Extraction of iodine from seaweed etc. C. SICOT and P. DAVION (F.P. 622,660, 4.2.26).—Seaweed is brought

into colloidal suspension or solution in water, the organic constituents are separated in the usual way, and the iodine is recovered from the mother-liquor by treatment with chlorine.

A. R. POWELL.

Recovery of organic substances containing iodine from marine algæ. P. GLOESS (G.P. 446,866, 4.9.23. Fr., 30.5.23. Cf. F.P. 578,564; B., 1927, 956).—The aqueous extract of dried marine algæ is treated with a solution of lead or copper acetates and the precipitate is separated and digested with sodium sulphide whereby a solution of the sodium salt of the organic iodine compound is obtained. The mother-liquor from the acetate treatment is utilised for the recovery of a potash fertiliser.

A. R. POWELL.

Recovery of bromine from iron bromide. KALIFORSCHUNGS-ANSTALT G.M.B.H., Assees. of F. KASELITZ and P. HÖFER (G.P. 449,736, 24.12.26).—Dry iron bromide, Fe_3Br_8 , is heated at about 300° in a current of air or in the presence of oxidising agents, *e.g.*, bromates.

L. A. COLES.

Sulphuric acid (G.P. 449,286). **Liquefying air** (B.P. 271,520).—See I. **Acetate liquor from wood** (G.P. 448,348). **Lead sludge** (U.S.P. 1,682,562).—See II. **Iodine compositions** (B.P. 267,557—8).—See XX.

VIII.—GLASS; CERAMICS.

Florida clay in bone china bodies. C. E. JACKSON (Trans. Ceram. Soc., 1928, 27, 151—152).—Florida clay was substituted in varying degrees for English china clay in a body made up of 50% of calcined bone, 20% of Cornish stone, and 30% of china clay. Samples were fired to cone 11 and with one exception were satisfactory. Best results were obtained with bodies containing 9—12% of English china clay and 18—21% of Florida clay. Characteristic properties of Florida clay are its plasticity and strength. It increases considerably the dry strength of a body, yet does not affect the colour of the fired body so much as ball clay.

F. SALT.

Refractories for the pottery industry. II. **Saggars.** W. EMERY (Trans. Ceram. Soc., 1928, 27, 169—219; cf. B., 1927, 44).—Data obtained from a number of saggars users indicated that losses in saggars were mainly due to cracking across the bottom and up the sides. (Information relating to the bone china and porcelain industries is not included.) A critical review of the literature is presented in relation to raw materials, their properties and methods of preparation, and to the manufacture of saggars. The results of a micro-examination of fired mixtures of siliceous fireclay and glazed grog, and of siliceous fireclay and fireclay grog, are given in an appendix.

F. SALT.

The system $\text{Al}_2\text{O}_3\text{--SiO}_2$ and its importance in connexion with ceramic materials. G. MALQUORI (Annali Chim. Appl., 1928, 18, 352—361).—A review of physico-chemical research on this system.

F. G. TRYHORN.

Determination of iron in silicates. A. E. J. VICKERS (Trans. Ceram. Soc., 1928, 27, 156—160).—Methods are described for determining both ferrous and ferric oxides in silicates, the data having been compiled from several sources and adapted to the special

requirements of clay analysis. Directions are included for the use of the Reinhardt method of titrating iron, and also of the colorimetric determination of iron by the thiocyanate method. F. SALT.

See also A., Oct., 1088, Hygroscopic water of clays (OKAZAWA). 1110, Weathering of clays (OKAZAWA).

PATENTS.

Forming ceramic ware. H. R. STRAIGHT (U.S.P. 1,682,200, 28.8.28. Appl., 14.6.26).—Granulated shale is piled in a vertical stack the bottom and sides of which are sealed, and while the central and bottom portions are stirred steam is introduced under pressure in quantities in excess of its condensation, reducing the shale to a plastic state. H. ROYAL-DAWSON.

Heat-treatment of ceramic articles. T. G. McDUGAL (B.P. 295,955, 21.2.27).—Sparkling-plug porcelains, tiles, electrical insulators, etc. are passed, preferably in single file, through a tunnel kiln by a conveyor outside the kiln with supporting members of a minimum size projecting into the furnace through a narrow slot in the wall, the width of the ends of the members being not greater than that of the slot. The walls of the kiln are shaped to focus the heat on to all parts of the surface of the articles. L. A. COLES.

Manufacture of artificial plagioclase compounds. F. SINGER (B.P. 282,402, 14.12.27. Ger., 17.12.26).—The oxides of bivalent metals (magnesium, barium, etc. and bivalent iron) are mixed with alumina and silica compounds (except alkali double silicates) in proportions corresponding approximately to the composition of natural plagioclase, and the mixture is heated at a temperature at least 50° below its fusion point. F. SALT.

Refractory body and method of making same. HARTFORD-EMPIRE Co., Assees. of P. G. WILLETTTS (B.P. 266,751, 28.2.27. U.S., 6.3.26).—A mixture of 4 pts. of raw aluminium hydrate (diaspore or bauxite), 4 pts. of calcined aluminium hydrate, 2 pts. of calcined alumina, 3 pts. of Grossalmerode (or similar) clay, and 5 pts. of a raw clay containing about 35% Al_2O_3 is ground to pass 325-mesh, made into a slip with water and agitated at about 90°, screened, filter-pressed, pugged, and finally dried. One portion of the dried mixture is converted into grog by calcination at 1750° and crushed to pass 12-mesh; the other is reground to 325-mesh. A mixture of these two portions, containing at least 50% of the grog portion, is then made into a paste with about 20% of water, pugged, partially dried, and moulded under pressure. The pressed blocks are fired at 1730°. [Stat. ref.] F. SALT.

Abrasive compositions. CARBORUNDUM Co., LTD. From H. R. POWER (B.P. 296,195, 30.8.27).—A bonding paste for abrasive grains of silicon carbide comprises fuller's earth, bentonite, etc., water, and material for retarding the evaporation of water, *e.g.*, maize syrup and glycerin. L. A. COLES.

Production of glass. M. THOMAS, Assr. to PATENT-TREUHAND GES. F. ELEKTR. GLÜHLAMPEN M.B.H. (U.S.P. 1,684,332, 11.9.28. Appl., 26.6.26. Ger., 1.8.25).—See B.P. 256,189; B., 1927, 109.

[Arrangement of] kilns [for burning bricks, tiles, etc.]. E. G. SPENCER-CHURCHILL (B.P. 296,844, 29.6.27).

Manufacture of splinterless glass sheet. P. J. PORT and J. NEWTON (B.P. 296,825, 13.6.27).

Sifting machines for treating potters' slip, powdered substances, etc. J. W. RATCLIFFE, and E. COTTON, LTD. (B.P. 296,959, 5.4.28).

Abrasive articles (B.P. 267,516).—See XIII.

IX.—BUILDING MATERIALS.

See also A., Oct., 1095, Decomposition of alkaline-earth sulphates (ZAWADSKI and others). System calcium oxide-silica-water (BAYLIS).

PATENTS.

Moulded articles such as tiles, slabs, etc. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of L. E. BARRINGER (B.P. 282,810, 29.12.27. U.S., 29.12.26).—Moulded articles suitable as building materials or in the construction of refrigerators comprise asbestos bonded with an inorganic binder, *e.g.*, hydrated calcium silicate, coated with a layer of a synthetic resin made from a polyhydric alcohol and a polybasic acid, *e.g.*, from glycerin and phthalic acid, and preferably mixed with fillers and plasticisers. L. A. COLES.

Materials for making tiles, slabs, road surfaces, etc. F. MORTON (B.P. 296,626, 28.1.28).—Coal-tar or blast-furnace pitch having a twisting point of 40–65° is melted and mixed with granite or other stone chip-pings and is moulded while hot, under pressure, with a heated coloured filler of powder or sand. W. G. CAREY.

Colouring mineral matter [slate]. II. C. FISHER, Assr. to RICHARDSON Co. (U.S.P. 1,680,941, 14.8.28. Appl., 25.11.22).—Crushed slate is impregnated with copper sulphate solution, dried, and heated at 800° to decompose the sulphate. The mass is then impregnated with ferrous sulphate and again calcined, whereby copper ferrite is formed on the surface of the slate particles, the colour of which is modified according to the quantities of the two sulphates used. A. R. POWELL.

Method of preserving wood and wood preservative. G. GUNN (U.S.P. 1,684,222, 11.9.28. Appl., 2.6.26. U.K., 26.3.26).—See B.P. 273,007; B., 1927, 655.

Tarry emulsions (B.P. 279,026).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Theory of blast-furnace smelting [of iron ores]. F. WÜST (Stahl u. Eisen, 1928, 48, 1273–1287; cf. B., 1928, 406).—The theory is based on analyses of iron pellets recovered from slag at various points in the furnace, on the composition of the slag, and on confirmatory tests carried out in the laboratory. The iron pellets suspended in the slag invariably contain higher percentages of impurities than does the pig iron tapped from the furnace, showing that the ore is reduced to iron above the tuyères and in passing through the

oxidising zone round the tuyères is subjected to a refining process in which part of the impurities is removed by oxidation similar to that taking place in a converter. Some granules of metal removed from a cold furnace just above the tuyères contained up to 60% Mn, 8% Si, and 5% P; these large amounts could not have been taken up by the iron during its passage through the short distance between the melting zone and the tuyères, and hence must have been taken up by cementation in the solid state. Laboratory tests showed that at 1100° in an atmosphere of hydrogen iron absorbed 8% Mn from manganese oxide in 7 hrs., at 1200° in an atmosphere of carbon monoxide it absorbed 3.8% Mn and 0.27% P, and, in contact with carbon at 1170° it absorbed 8% P, 5% Mn, and 2.5% Si in 4–5 hrs. from their respective oxides.

A. R. POWELL.

Dependence of the operation of the Thomas converter on the temperature curve. R. FRERICH (Stahl u. Eisen, 1928, 48, 1233–1240).—From measurements made with the Holborn-Kurlbaum ardrometer temperature-time curves have been constructed for numerous charges of cast iron blown in the Thomas converter under varying conditions of wind pressure. When a steady even pressure is maintained throughout the blow the points at which the silicon, carbon, and phosphorus begin to burn are plainly visible on the temperature curve. The curve rises more steeply at the beginning and end of the operation than in the middle owing to the greater heat of combustion of silicon and phosphorus compared with that of carbon. By varying the pressure of the blast the temperature at any stage of the blow can be regulated as desired, but the best results are obtained by maintaining throughout the blow as even and as high a pressure as is possible without causing iron to be projected out of the converter. In this way the ferrous oxide content of the slag may be kept low with a consequent improvement in the quality of the steel and the removal of any fear of red-shortness in the finished metal owing to the greater ease with which complete deoxidation may be effected. A high, even pressure of blast also reduces the time required in blowing, lowers the amount of corrosion of the lining per charge, decreases the nitrogen content of the steel, and increases the yield from a charge owing to the smaller loss by burning the iron.

A. R. POWELL.

Comparative tests on the mechanical properties of cast steel at elevated temperatures. A. POMR (Stahl u. Eisen, 1928, 48, 1321–1330).—The mechanical properties of eight steels from the Siemens-Martin furnace, three from the Bessemer converter, and one from the electric furnace have been determined at temperatures between 20° and 500°. The elastic limit and yield point decreased almost linearly with rise of temperature, and at 500° were 42–67% below the values at 20°, steels with a high manganese content giving the highest values at 500°. Of the open-hearth steels tested, that with 1.65% Ni had a relatively low yield point (9.8 kg./mm.²) at 500°, but that with 2.68% Ni had a high yield point (30.5 kg./mm.²) at 20°. Of the Bessemer steels those with a relatively high phosphorus and sulphur content behaved

just as satisfactorily as those with only small quantities of these impurities. With rise of temperature the tensile strength of all the steels decreased slightly up to 100°, then increased again to a maximum between 200° and 300°, after which a rapid fall in strength took place, the values at 500° being 45–68% below those at 20°. The decrease in strength was least marked with steels having a high manganese or carbon content; nickel had little effect in maintaining the strength at high temperature, but phosphorus and sulphur up to 0.15% increased the hot strength of Bessemer steels. The elongation and reduction in area of all the steels decreased to a minimum at 200–300°, and then increased rapidly. The impact strength increased more or less steeply according to the composition to a maximum at 100–200°, then fell slowly to 300–400°, and then rapidly. The values of the impact strength are shown to be dependent on the size of the test-piece and on its position in the casting.

A. R. POWELL.

Conditions of formation of cementite. G. CHARPY and P. PINGAULT (Compt. rend., 1928, 187, 554–556).—The formation of cementite by the carburisation of hydrogen-free electrolytic iron filings, heated in contact with hydrocarbons or alkali cyanides, has been followed from changes in density and magnetic properties. After complete transformation, the filings, which contained 6.75% C, were capable of scratching glass and dissolved completely in nitric acid leaving no trace of graphite. The cementite remained stable when heated *in vacuo* for 10 hrs. at 1000°, and apart from slight variations due probably to unchanged iron, the heating curves showed no marked critical point.

J. GRANT.

Use of aluminium, steels, VA alloys, and silicon alloys in the nitric acid industry. B. WAESER (Chem. Fabr., 1928, 529–530, 544–546).—A review of the properties, uses and resistance to corrosion of aluminium, chromium-nickel steels, and silicon-iron alloys (Thermisilid, Tantiron, and Duriron), with special reference to their behaviour in nitric acid of varying concentrations.

A. R. POWELL.

Smelting copper in the reverberatory furnace. G. L. OLDRIGHT and F. W. SCHROEDER (Amer. Inst. Min. Met. Eng. Tech. Pub., 1928, No. 49, 20 pp.).—The charge is finely divided and is preheated, and powdered coal or oil is used as fuel. Preheating the air, and extending the length and raising the temperature of the smelting zone are suggested. The condition of the brick lining is described; water-cooling the silica bricks is not feasible.

CHEMICAL ABSTRACTS.

External corrosion of copper and brass service pipe [for water]. K. H. LOGAN and S. P. EWING (J. Amer. Water Works' Assoc., 1928, 20, 390–403).—Specimens of brass buried in different types of soil for upwards of two years indicated on examination that although the losses were too small and variable to permit definite conclusions, brasses of high copper content withstood soil corrosion well and were definitely superior to ferrous materials. There was little indication that galvanic action due to contact with lead or steel was a cause of accelerated corrosion. A serious cause of service-pipe failure in the neighbourhood of

powerhouses or sub-stations is due to stray electric currents passing from the pipe to the surrounding soil. The character of the pipe material is not a vital factor, though brasses with 30–40% Zn showed evidence of selective corrosion analogous to pitting in steel. The life of a service pipe under such conditions depends largely on the thickness of the pipe wall.

C. JEPSON.

Nickel mirrors by the nickel carbonyl method. C. G. FINK and W. G. KING, JUN. (Amer. Electrochem. Soc., Sept., 1928. Advance copy. 4 pp.).—By admitting nickel carbonyl vapour suddenly into an evacuated vacuum flask uniformly heated in an air oven to 180–200°, the walls may be coated with a bright, adherent, uniform deposit of nickel, which has a reflectivity for heat waves nearly as high as that of silver. The presence of traces of air, oxygen, chlorine, etc. is to be avoided as it leads to dark deposits. Preliminary flushing with hydrogen is effective in ensuring bright deposits, but is unnecessary if the vessel is evacuated to 5–10 microns pressure. Coatings of nickel on porcelain and metals may be similarly produced and serve as a foundation for the electrodeposition of other metals.

H. J. T. ELLINGHAM.

Corrosion in the tin can. I. Electrochemical relations of iron and tin. R. H. LUECK and H. T. BLAIR (Amer. Electrochem. Soc., Sept., 1928. Advance copy. 23 pp.).—In approximately 0.2*N*-solutions of various acids, having p_H values ranging from 0.81 to 2.98, iron dissolves with evolution of hydrogen but tin is unattacked. With an iron-tin couple hydrogen was evolved but the rate of evolution was always less than that from uncoupled specimens of iron. Experiments in a citric-malic acid solution of p_H 2.98 showed that this protective action of tin on iron was exhibited when the solution was aerated as well as when it was kept saturated with hydrogen or nitrogen. Corrosion was most rapid in the aerated solutions, but in all cases it is the tin which dissolves more rapidly from the couple. Cadmium coupled with iron protected it to a similar extent but the cadmium dissolved more rapidly than tin under the same conditions. Measurements of the *E.M.F.* of cells of the type Fe|canned foods|Sn show that, although tin is definitely cathodic to iron on open circuit, on short-circuiting, the *E.M.F.* falls rapidly and changes sign, the tin becoming the anode. Removal of either electrode and exposing it to the air gave it a more noble potential. With similar cells using iron and cadmium electrodes the latter was always the anode and the *E.M.F.* changed very little on short-circuit; using tin and cadmium, cadmium was again always anodic but the *E.M.F.* fell notably on closed circuit and recovered gradually when the circuit was opened. In a diaphragm cell containing a citric-malic acid mixture buffered to p_H 3.5, the effect of saturating the electrolyte around the individual electrodes with oxygen or hydrogen was examined. With the iron in a solution saturated with oxygen or hydrogen, the tin electrode always became the anode whether the solution into which it dipped was kept saturated with oxygen or with hydrogen or not treated with gases at all. But with no gas passed through the solution around the iron, the tin was always cathodic if the solution into which it dipped was kept

saturated with oxygen or with hydrogen. Addition of hydrogen peroxide to the solution around the tin also kept it cathodic to the iron. These results are considered to be related to the high hydrogen overvoltage of tin compared with that of iron; the tin becomes polarised and then hydrogen is more readily liberated at the iron. The abnormal tendency of red fruits to cause perforation of tin cans cannot be explained on current views. It is possible that mild oxidising agents are present and serve to cause the microscopic areas of exposed iron to become anodic to the surrounding tin.

H. J. T. ELLINGHAM.

Porosity of electro-deposited chromium. E. M. BAKER and A. M. RENTE (Amer. Electrochem. Soc., Sept., 1928. Advance copy. 9 pp.).—Chromium was deposited on polished nickel cathodes from baths containing chromic acid and sulphuric acid in the molar concentration ratio of 100:1, and the porosity of the plating was determined by cathodic treatment of the plates in an acid copper sulphate bath. Copper only deposits at points where the underlying nickel is exposed. For all bath compositions investigated at temperatures from 35° to 60° minimum porosity was attained at a certain thickness of deposit ($< 4 \times 10^{-3}$ mm.). At 45° this minimum porosity becomes progressively lower as the total bath concentration is increased. With a bath which is 2.5*M* to chromic acid, the least porous deposits are obtained at about 55°, the optimum thickness then being about 9×10^{-4} mm. For deposits 3×10^{-4} mm. thick, the porosity does not vary much with temperatures between 40° and 60°. But as the temperature or total concentration is increased it is necessary to increase the thickness of the deposit in order to get the minimum porosity obtainable at that temperature or concentration. The nature of the porosity has been studied and an explanation of it is suggested.

H. J. T. ELLINGHAM.

See also A., Oct., 1077, **X-Ray analysis of Heusler's alloy** (PERSSON). 1078, **X-Ray analysis of silver-aluminium alloys** (WESTGREN and BRADLEY). **Changes in the alloy CuAu** (GORSKY). 1079, **Slip-bands in stretched aluminium crystals** (YAMAGUCHI). 1080, **Mechanical properties of brass crystals** (MASIMA and SACHS). 1081, **Magnetic properties of cobalt** (SAMUEL). **Magnetisation of nickel crystals** (KAYA). 1082, **Iron crystals** (DUSSLER). **Superconductivity** (KRETZSCHMANN). "Resistance limits" for gold-copper system (LE BLANC and others). 1083, **Electrical resistance of molten metals** (MATSUYAMA). **Conductivity of metals** (LAPINSKY). **Volume change of manganese during solidification** (MATSUYAMA). 1085, **Lead-antimony alloys** (BRONIEVSKI and SLIVOVSKI). **Palladium antimonides** (THOMASSEN). 1094, **System antimony-arsenic** (MANSURI). **System copper-silicon** (MATSUYAMA). 1095, **System aluminium-calcium** (MATSUYAMA). 1098, **Passivity of chromium and its alloys** (STRAUSS and HINNÜBER). 1104, **Reactions in the evolution flask** (KITASHIMA). 1108, **Test for silver** (FEIGL).

Mischmetal as rectifier. KREMERS and THOMAS. **Co-deposition of copper and graphite.** FINK and PRINCE. **Nickel plating solutions.** DORRANCE and

GARDINER. **Electrodeposited nickel.** MACNAUGHTAN and HOTHERSALL. **Chromium plating.** BAKER and PETTIBONE.—See XI.

PATENTS.

[**Blast-furnace**] tuyère. G. W. VREELAND (U.S.P. 1,682,816, 4.9.28. Appl., 8.7.27).—The tuyère has a cylindrical water-cooled body slightly tapering towards the nose portion which is solid and is composed of a shell within which is a cast-iron ring. A. R. POWELL.

Regenerative open-hearth furnace. S. J. CORT, Assr. to BETHLEHEM STEEL CO. (U.S.P. 1,683,656, 11.9.28. Appl., 21.12.23).—The furnace is diabolo-shaped in plan and a system of buckstaves, tie rods, and channel members for supporting the roof is described.

B. M. VENABLES.

Melting furnaces for liquid fuel. G. REGNAC-PAILLE (B.P. 282,627, 15.8.27. Belg., 23.12.26).—The combustion chamber is provided with a tortuous passage which gradually narrows towards the fusion chamber and is so constructed as to present a deflecting elbow against which the mixture of air and fuel impinges and past which the flame, but not the liquid fuel, can pass into the fusion chamber.

M. E. NOTTAGE.

Manufacture of cast iron. A. F. MEEHAN, Assr. to MECHANITE METAL CORP. (U.S.P. 1,683,086—7, 4.9.28. Appl., [A] 16.6.27, [B] 27.8.27).—(A) Molten grey cast iron is treated with a silicide of an alkaline-earth metal and a graphitising agent such as nickel. (B) Molten iron which, if cast, would contain more than 2% of combined carbon is treated with such a proportion of calcium as will produce the degree of graphitisation required (cf. B.P. 210,118; B., 1924, 262).

A. R. POWELL.

Improvement of fused material, especially cast iron. MASCHINENFABR. OERLIKON (Swiss P. 120,564, 14.7.26).—The fused material is stirred by means of agitators constructed of highly refractory material covered with a high-melting metal.

L. A. COLES.

Production of iron sponge. S. E. SIEURIN (B.P. 296,235, 6.12.27).—Iron sponge, produced by imbedding iron ore in a solid reducing agent (carbon) mixed with lime or magnesia, and reducing in the absence of air at a temperature below the m.p., may be made free from sulphur by using the reducing agent in such an excess over the amount necessary for the reduction of the ore that carbon still remains in the ash at the end of the process. The cooling of the product is accelerated inversely as the amount of excess of reducing agent, so that the calcium or magnesium sulphide formed in the charge is prevented from decomposing.

M. E. NOTTAGE.

Improving the tensile qualities of drawn steel wire. VEREIN. STAHLWERKE A.-G. (B.P. 272,240, 7.6.27. Ger., 4.6.26).—After cold-drawing, the wire is tempered at a temperature between 350° and the A₁ point either by drawing it through a lead or salt bath or by heating the whole coil in an oven. The temperature of tempering is increased to a small degree as the carbon content increases, whilst the duration is extended approximately in proportion to the square of the radius of the wire so that the capacity for expansion increases.

This heat-treatment precedes any galvanising, tinning, or metal-coating process.

M. E. NOTTAGE.

Treating [annealing] strip metal [iron or steel]. W. E. WATKINS (B.P. 279,787, 5.7.27. U.S., 28.10.26).—Iron or steel strip is passed continuously through a horizontal annealing furnace through which hot reducing gases are passed. To prevent oxidation the metal may previously be coated with an oil having an asphaltic base or with a mixture of a reducible metal oxide and the oil so as to produce a metallic coating on the strip. The treated strip passes from the furnace, through a cooling chamber filled with non-oxidising gases, thence into a pickling bath, washing bath, and drying apparatus in succession.

A. R. POWELL.

Removal of iron from minerals [bauxite etc.]. I. G. FARBENIND. A.-G. (Swiss P. 120,854, 25.9.25. Ger., 1.10.24).—The minerals are treated with carbon monoxide at 250° under pressure to yield volatile iron carbonyls.

L. A. COLES.

Precipitation (cementation) of copper. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 296,814, 9.6.27).—In the precipitation of metallic copper from solutions by means of iron, a gaseous agitating agent is employed which contains oxygen and such a quantity of sulphur dioxide as will prevent re-oxidation of the metallic copper produced.

M. E. NOTTAGE.

Deoxidation of metals or alloys. SIEMENS & HALSKE. A.-G. (B.P. 288,543, 22.2.28. Ger., 11.4.27).—Copper and nickel or their alloys are deoxidised by the addition of up to 0.5% of beryllium in the form of a 5–10% alloy with the copper or nickel or both.

A. R. POWELL.

[**Non-oxidisable**] alloys. E. MANOS (B.P. 282,096, 10.12.27. Switz., 11.12.26).—Hard white alloys containing 72–74% Cu, 18–20% Ni, 6–7% Zn, 0.1–1% Al, 0.025–0.2% Fe, 0.01–0.2% Si, and 0.05–0.3% Mg are claimed. A particular alloy consists of 72.9% Cu, 19.6% Ni, 0.3% Al, 6.86% Zn, 0.1% Fe, 0.14% Si, and 0.1% Mg.

F. G. CROSSE.

Alloy. S. E. WINSLOW, Assr. to CONSOL. ASHCROFT HANCOCK CO. (U.S.P. 1,683,749, 11.9.28. Appl., 4.3.24).—In a nickel-copper-tin alloy, the respective metals are present in the atomic proportions of 10 : 4 : 1 or less.

F. G. CROSSE.

Refinement of nickel alloys. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (B.P. 296,112, 25.4.27).—The sulphur content of alloys containing not less than 50% Ni may be minimised or eliminated by melting the metal or scrap in the presence of slag in a reducing atmosphere, preferably in an induction furnace. The slag may consist mainly of lime and silica together with a small amount of either fluorspar or calcium chloride. After the supply of reducing gas is cut off, the slag is removed from the surface and a small amount of the oxide of one of the constituents of the alloy is introduced to remove the absorbed reducing gases.

M. E. NOTTAGE.

Fusible lead alloys. E. C. R. MARKS. From AMER. MACHINE & FOUNDRY CO. (B.P. 296,584, 20.10.27).—An alloy for use as a solder consists of pure lead together

with 0.05–0.10% of lead phosphide and, if desired, 1% of a brightening metal such as aluminium, antimony, tin, or zinc. M. E. NOTTAGE.

Separating impurities from impure lead or lead alloys. H. HARRIS (B.P. 296,557, 3.9.27).—Arsenic, tin, and antimony are removed successively from impure lead by allowing the molten lead to fall in fine streams into a molten mass of sodium hydroxide, with or without sodium chloride, to which regulated amounts of litharge or lead oxide dross are added. For the removal of arsenic and tin from antimonial lead, antimony oxide may be used in place of some or all of the lead oxide.

A. R. POWELL.

Production of aluminium castings. H. MAURER, JUN. (Swiss P. 120,859, 1.6.26).—Compact castings free from blow-holes are obtained by stirring the aluminium just at its m.p. with 2 pts. of powdered white glass per 100 pts. of metal, and removing the impurities separating at the top and bottom of the melt. L. A. COLES.

Bearing-metal alloys. T. GOLDSCHMIDT A.-G. (B.P. 295,265, 25.2.27. Ger., 25.2.26. Cf. B.P. 266,696; B., 1928, 789).—Aluminium may be rendered suitable for bearing purposes by the addition of one or more of the metals calcium, barium, strontium, or beryllium and incorporating with the alloys thus produced one or more of the elements titanium, vanadium, molybdenum, tungsten, chromium, iron, manganese, or boron which do not form with aluminium either solid solutions or eutectic mixtures but which crystallise out as such, forming hard crystals in the alloy. One or more of the metals magnesium, zinc, copper, cobalt, nickel, or cerium, also silicon or silicides of lithium and magnesium, may also be introduced as additional components.

M. E. NOTTAGE.

Resistance alloys. BRIT. THOMSON-HOUSTON Co., LTD., Assees. of T. S. FULLER (B.P. 285,908, 20.2.28. U.S., 25.2.27).—An aluminium alloy containing 4–10 Mn and 2–8% Zn and preferably consisting of 90% Al, 6% Mn, and 4% Zn has a resistance of about 11.5 microhms per c.c.

M. E. NOTTAGE.

Making metallic tungsten. K. ANJOW, ASSR. to MITSUBISHI KOGYO KABUSHIKI KAISHA (U.S.P. 1,682,058, 28.8.28. Appl., 3.7.26. Japan, 18.5.26).—Technically pure tungsten is obtained by heating the crude metal with alkali solution in a closed vessel under pressure.

H. ROYAL-DAWSON.

Form of tungsten and manufacture of same. C. G. FINK (U.S.P. 1,675,486, 3.7.28. Appl., 16.1.24).—Tungsten slugs are mixed with a reducing agent and heated in hydrogen at high temperatures to remove the intercrystalline impurities by reduction. A tungsten which is largely crystalline results. C. HOLLINS.

Treatment of ores etc. containing platinum. S. C. SMITH (B.P. 296,744, 7.6.27).—Sulphide ores or concentrates containing the platinum metals in association with copper, nickel, and iron sulphides are smelted to obtain a copper-nickel-iron matte. This is crushed to 60-mesh and digested at 70–80° with sulphuric acid (400 g./litre), whereby the iron and nickel sulphides are dissolved, leaving a residue of copper sulphide and precious metals. This residue is roasted and leached with

dilute sulphuric acid to obtain a rich concentrate from which the platinum metals may be recovered by known means. A. R. POWELL.

Increasing the conductivity of metals. S. RUBEN (U.S.P. 1,683,209, 4.9.28. Appl., 15.2.26. Renewed 12.5.27).—Metal wires are heated by resistance while passing through an electromagnetic field.

A. R. POWELL.

Coating of metals by the cementation process. METALLBANK U. METALLURGISCHE GES. A.-G. (B.P. 272,920, 15.6.27. Ger., 15.6.26).—Iron, copper, and other metals or alloys are provided with a coating of aluminium by heating them in an inert atmosphere at 700–1200° embedded in a mixture of aluminium dross containing 15–30%, preferably 20%, of free aluminium capable of diffusing into the surface of the metal under treatment.

A. R. POWELL.

Material for use in case-hardening metals. A. E. TAYLOR, H. W. BARTON, and F. A. JONES (B.P. 297,163, 25.6.27).—The material comprises disintegrated Dum Palm nut, Coquilla nut, apple nut, or Coroza nut, or mixtures of these, which may be dried or heat-treated, and barium carbonate, with or without the addition of charcoal and sodium carbonate.

L. A. COLES.

Production of iron castings with a low carbon content. K. EMMEL and H. WALBERT (U.S.P. 1,683,714, 11.9.28. Appl., 11.8.25. Ger., 9.12.24).—See B.P. 244,405; B., 1927, 79.

Manufacture of steel. R. A. HADFIELD (U.S.P. 1,683,886, 11.9.28. Appl., 20.11.22. U.K., 19.6.22).—See B.P. 204,110; B., 1923, 1182 A.

Casting articles of corrosion-proof steel. F. HAUPTMEYER, ASSR. to F. KRUPP A.-G. (U.S.P. 1,684,700, 18.9.28. Appl., 26.1.27. Ger., 18.2.26).—See B.P. 266,305; B., 1927, 753.

Flux for welding or soldering of aluminium and other metals. W. REUSS (B.P. 296,923, 10.11.27).—See U.S.P. 1,675,664; B., 1928, 610.

Flotation process. A. H. FISCHER, ASSR. to GUGGENHEIM BROS. (U.S.P. 1,684,536, 18.9.28. Appl., 24.1.27).—See B.P. 284,198; B., 1928, 373.

Jigs for washing ores etc. T. V. O'HARE (B.P. 296,889, 26.8.27).

Centrifugal casting of hollow metal bodies. A. POSSENTI and C. SCORZA (B.P. 296,902, 28.9.27. Addn. to B.P. 277,996).

Annealing of metal bands. O. BUSSE, and MANSFELD A.-G. F. BERGBAU U. HÜTTENBETRIEB (B.P. 296,146, 11.6.27).

Production of copper cakes. G. B. ELLIS. From ANACONDA COPPER MINING Co. (B.P. 296,746, 7.6.27).

Manufacturing compound metal sheets. F. KRUPP A.-G. (B.P. 280,518, 21.10.27. Ger., 11.11.26).

Apparatus for atomising and spraying metals and other fusible materials [in the form of wire or powder]. METALLISATOR BERLIN A.-G., and E. GREISER (B.P. 296,546, 24.8.27).

Ferrosilicon (B.P. 290,971).—See VII.

XI.—ELECTROTECHNICS.

High-frequency induction furnace for chemical preparations above 1000°. C. N. SCHUETTE and C. G. MAIER (Amer. Electrochem. Soc., Sept., 1928. Advance copy. 14 pp.).—A special furnace was constructed in which the inductor coil had 2.25 turns/cm., the turns being insulated from each other with asbestos paper coated with Bakelite varnish. In this furnace a graphite cylinder was heated to 1300° in 5 min., whereas with a similar furnace with 0.79 turn/cm. it required 14 min. when operated at the same power. In order to raise the cylinder to 1900°, 10 kw. sufficed with the former furnace, and 14 kw. with the latter. For heating *in vacuo*, crucibles were supported in specially designed silica tubes. To avoid imbedding the crucibles in magnesia or other insulator a radiation screen was devised and was effective in preventing overheating of the silica tube. A satisfactory method of preparing silica crucibles by heating a mixture of raw and pre-burned silica in graphite moulds in the high-frequency furnace has been developed, and the technique of the operation is described. Sintered magnesia crucibles have also been made by a similar method. In the preparation of ferrous oxide by heating iron and magnetite *in vacuo* in iron crucibles local melting of the crucibles occurred. This was avoided by using a series of superimposed iron trays with holes drilled in the centre so that the oxide flowed from one tray to the others. At higher temperatures rotation of the whole vacuum tube permitted operation in an iron crucible imbedded in magnesia, even when the iron was molten. The melting of copper-iron alloys in hydrogen and the preparation of pure ferrous sulphide are also referred to.

H. J. T. ELLINGHAM.

Thermoelectric measurement of temperatures above 1500°. H. L. WATSON and H. ABRAMS (Amer. Electrochem. Soc., Sept., 1928. Advance copy. 13 pp.).—The construction and characteristics of a tungsten-graphite thermocouple are described. It has an accuracy of about $\pm 10^\circ$ up to 1600°, and with suitable protection can be used up to much higher temperatures. Short runs at temperatures over 2300° have been made, but the life of the couple is short under such conditions. It can be used under conditions where platinum-rhodium couples would fail through contamination. Special refractory sheath materials are being developed to suit the numerous possible applications of the thermocouple, such as in the manufacture of steel, glass, carbide, etc. An advantage of its use for such purposes is that it lends itself readily to automatic recording and controlling devices which otherwise could not be employed at such high temperatures.

H. J. T. ELLINGHAM.

Effect of adsorbed gases on the contact resistance of carbon. R. H. WRIGHT and M. J. MARSHALL (Amer. Electrochem. Soc., Sept., 1928. Advance copy. 12 pp.).—In a specially designed apparatus two carbon filaments arranged at right angles to one another could be brought into contact and pressed together with any desired pressure. The decrease of the electrical resistance of the contact with increase of contact pressure was measured both in the presence of air and in a vacuum after heating the filaments to a high temperature *in*

vacuo to remove adsorbed gas. At low contact pressures the contact resistance is lower when the adsorbed gas film has been removed, but at high pressures, when the contact area is doubtless much larger owing to the elasticity of the material, the resistance is practically the same as in air.

H. J. T. ELLINGHAM.

Conductivity of organic solvents. D. B. KEYES, S. SWANN, JUN., and H. W. HOERR (Amer. Electrochem. Soc., Sept., 1928. Advance copy. 8 pp.).—By means of a simple direct-current method the approximate specific conductivities of a number of commercial samples of organic liquids and mixtures have been measured at 25°. Data obtained by previous workers for common organic liquids are tabulated.

H. J. T. ELLINGHAM.

Voltaic hydrogen generator. W. C. GARDINER and G. A. HULETT (Amer. Electrochem. Soc., Sept., 1928. Advance copy. 10 pp.).—The voltaic cell consisting of an amalgamated zinc anode and a platinum-black or nickel cathode in a solution of sodium hydroxide containing a little barium hydroxide has been adapted for use as a generator to furnish a slow stream of pure hydrogen over a long period. The rate of hydrogen evolution may be controlled by introducing a variable resistance in the circuit; but no impurities are introduced into the hydrogen when the generator is run on a short-circuit. No oxygen is produced by the action of the cell, and the generator is designed so as to deliver pure hydrogen at once. Freedom from oxygen was indicated by the use of luminescent bacteria (cf. A., 1923, i, 1267).

H. J. T. ELLINGHAM.

Use of mischmetal as an electrolytic rectifier. H. C. KREMERS and D. C. THOMAS (Amer. Electrochem. Soc., Sept., 1928. Advance copy. 6 pp.).—A cell consisting of a lead electrode and an electrode of mischmetal, specially prepared so as to be free from iron, in solutions of sodium phosphates or sodium tungstate was examined with respect to its rectifying properties. An electrolyte containing 25% of trisodium phosphate and 10% of sodium hydroxide gave most satisfactory results. The film forms rapidly and is permanent, and the degree of half-wave rectification approaches that of a tantalum cell. Break-down voltages as high as 90 volts were obtained.

H. J. T. ELLINGHAM.

Co-deposition of copper and graphite. C. G. FINK and J. D. PRINCE (Amer. Electrochem. Soc., Sept., 1928. Advance copy. 6 pp.).—Experiments have been made to determine whether a self-lubricating bearing metal could be made by simultaneous electro-deposition of copper and graphite from acid copper sulphate solutions containing "aquadag." With a solution containing 200 g. of copper sulphate crystals, 100 g. of sulphuric acid, and 6.55 g. of graphite per litre at 20° and 2.08 amp./dm.² a fine-grained adherent deposit containing 11% of graphite was obtained on the cathode. Increasing the graphite concentration in the solution above 13 g./litre does not increase the amount of it in the deposit. The deposits were brittle even when the bath temperature was raised to 55°, but the brittleness can be largely removed by annealing at a red heat. At 55° the deposits were coarser grained than at 20° but adhered well. Since graphite settles out

rapidly from acid solutions, gelatin was tried as a protective agent, but at 55° it gives rise to non-adherent deposits. Vigorous stirring is an effective substitute for addition of gelatin at 55°. H. J. T. ELLINGHAM.

Polarisation and resistivity in nickel-plating solutions. R. L. DORRANCE and W. C. GARDINER (Amer. Electrochem. Soc., Sept., 1928. Advance copy. 10 pp.).—To *N*-nickel sulphate, 0.25*M*-boric acid solutions were added varying concentrations of chlorides of ammonium, sodium, potassium, magnesium, or nickel, and the influence of these chlorides on the anodic and cathodic polarisations and on the resistivity of the solution at 17° was determined by means of the Haring cell (B., 1926, 497). The p_H value of all the solutions was adjusted to 5.6 ± 0.2 . Addition of any of the above chlorides at a concentration of 0.25*M* reduced the anodic polarisation at 1 amp./dm.² from 1.78 to 0.3–0.5 volt, but increased the cathodic polarisation from 0.56 to 0.7–0.9 volt, whilst the resistivity fell from 10 ohm-cm. to 5–6, except with nickel chloride, when the value was 7.7. It is concluded that the cation associated with the chloride ion has no specific effect on the anodic polarisation. Further experiments showed that the bromide ion reduces anodic polarisation as effectively as the chloride ion, but the fluoride ion has no effect of this kind. Hydrogen peroxide does not affect the polarisations, but sodium citrate increases the anodic polarisation. Cadmium chloride gives a low cathodic polarisation and hence may be expected to lower the throwing power of the bath.

H. J. T. ELLINGHAM.

Causes and prevention of pitting in electro-deposited nickel. D. J. MACNAUGHTAN and A. W. HOTHERSALL (Trans. Faraday Soc., 1928, 24, 497–509).—Observations made of a magnified image of an illuminated cathode surface during deposition indicate that pits, circular or tailed, are caused by bubbles of gas, usually hydrogen, clinging to the surface during deposition, or by a succession of bubbles from a point on the surface. That tailed pits were probably due to local stagnation of electrolyte, and consequent depletion in metallic ions immediately above a bubble was shown by comparison with pits artificially produced by screening the surface with small glass beads. Pitting was less frequent when the anodes and solutions were replaced by highly purified materials, and was localised to defects in the cathode surface where bubbles formed. Bubbles and pitting were both absent in the case of a ground and polished copper surface or of a copper surface the defects of which had been covered by a preliminary deposit of copper. Hence, to remove these defects, a steel surface was given first a thin nickel deposit, then one of copper from acid copper sulphate, and a final nickel deposit. Preferential discharge of hydrogen at surface defects suggests decreased hydrogen overvoltage relative to elsewhere on the surface. The effect of deposited copper, therefore, is to cover surface defects, thus eliminating areas of lower overvoltage and providing basis metal for nickel deposition upon which overvoltage of hydrogen discharge is higher than upon steel. Use of commercial materials with copper-treated basis metal did not invariably prevent pitting, which was attributed to a defective condition of the solution. N. M. BLIGH.

Steel anodes for chromium plating. E. M. BAKER and E. E. PETTIBONE (Amer. Electrochem. Soc., Sept., 1928. Advance copy. 4 pp.).—With a bath containing 2.5 mols. of chromic acid and 0.025 mol. of sulphuric acid per litre at 44–46° the rate of corrosion of steel anodes increases rapidly with increase of carbon content of the steel; a very low rate of corrosion is found with electrolytic iron anodes. For a given anode material the rate of corrosion is practically proportional to the anodic current density over the range 3.6–21.6 amp./dm.², and at given current density it decreases with time of electrolysis, the decrease being most marked in the case of anodes with the highest carbon content. The steady value of the ratio of tervalent to sesquivalent chromium which is established in the solution during electrolysis is not appreciably affected by varying the carbon content of the anodes from 0 to 1%.

H. J. T. ELLINGHAM.

See also A., Oct., 1101, **Preparation of cuprous oxide** (ABEL and REDLICH). 1102, **Oxidation of organic substances** (MARIE and LEJEUNE). 1149, **Furnace for organic combustions** (PHILLIPS and HELLBACH).

Hot centrifuge. HÜTTER.—See I. **Insulating oils.** ZIMMERMANN.—See II. **Titration of hypochlorite etc.** RIUS and ARNAL.—See VII. **Corrosion of tin cans.** LUECK and BLAIR.—See X.

PATENTS.

Electric furnace. H. GEORGE (B.P. 279,818, 10.10.27. Fr., 27.10.26).—A refractory crucible is surrounded by an outer crucible, and the intervening space contains tin which is heated by an electric current.

J. S. G. THOMAS.

Induction pressure or vacuum furnace. E. F. NORTHRUP, Assr. to AJAX ELECTROTHERMIC CORP. (U.S.P. 1,683,986, 11.9.28. Appl., 1.8.25).—In a furnace having top and bottom walls and an inductor side wall, all vacuum-sealing against passage of air, the inductor side wall comprises the sealing part of the side of the enclosure.

J. S. G. THOMAS.

Electrolytic rectifier. H. C. KREMERS (U.S.P. 1,682,846, 4.9.28. Appl., 16.7.26).—The cell comprises a filming electrode containing a rare-earth metal, e.g., cerium, and at least one non-corrodible metal electrode.

F. G. CLARKE.

Electric [storage] batteries. SILICA GEL CORP., Assees. of W. J. PLEWS (B.P. 272,916, 14.6.27. U.S., 15.6.26).—Plate electrodes are supported in spaced relation by a non-yielding packing of hard, adsorbent, granular or coarsely-ground silica gel, with or without electrolyte.

J. S. G. THOMAS.

Sulphur-proofing metallic conductors for cables etc. F. KIRSCHNER and J. HESS (B.P. 297,161, 23.6.27).—Zinc is electrolytically deposited on a coating of an easily fusible metal or alloy, e.g., tin, nickel, or tin alloy, which is itself deposited upon the conductor and fused; a thin layer of tin is then deposited on the zinc.

J. S. G. THOMAS.

Coatings conductor of electricity for electro-plating non-metallic articles. K. BARANOVSKI (B.P. 297,260, 21.12.27).—Water is added from time to

time in small quantities to a syrupy paste produced by boiling finely-powdered carbon, soot, or synthetic graphite with a dilute alcohol or ether; after heating until the alcohol or ether has been completely evaporated, the aqueous paste is mixed with soluble glass.

J. S. G. THOMAS.

Apparatus for electrical precipitation of suspended particles from gases. LODGE-COTTRELL, LTD. From LURGI APPARATEBAU GES.M.B.H. (B.P. 296,956, 26.3.28).—Resistances are inserted in the feeders of different groups of electrodes connected to a common source of high-tension unidirectional current, and/or between the discharge electrodes and the opposite collecting electrodes.

J. S. G. THOMAS.

Increasing the electric disruption strength or resistance of films consisting of cellulose esters or ethers. M. OW-ESCHINGEN (B.P. 281,663, 29.11.27. Austr., 1.12.26).—The dielectric character of the films is increased by soaking them with an oil of high disruptive strength (e.g., paraffin oil, transformer oil, etc.). The process is carried out *in vacuo* and subsequently under a pressure above atmospheric. F. G. CROSSE.

[Electrode mounting for] electric furnaces. Soc. ELECTRO-MÉTALLURGIQUE DE MONTRICHER (B.P. 282,747, 17.12.27. Fr., 28.12.26).

Separators for electrodes of electrolytic cells. J. L. WOODBRIDGE (B.P. 297,246, 23.11.27).

Electric incandescence lamps with concentrated filaments. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 297,170, 4.7.27).

Electrical heating apparatus for electroplating baths. H. ZANDER (B.P. 297,225, 1.10.27).

Carbon (B.P. 274,883).—See II. **Hydrogen and oxygen** (G.P. 449,603).—See VII. **Resistance alloys** (B.P. 285,908). **Conductivity of metals** (U.S.P. 1,683,209).—See X. **Sugar juice** (F.P. 618,779).—See XVII. **Antirachitic preparations** (B.P. 286,665).—See XX.

XII.—FATS; OILS; WAXES.

Electrically-heated apparatus for fat and oil Soxhlet extractions. F. GOGOLEV (Masloboino Zhir. Delo, 1928, No. 3, 18—19).—The usual apparatus is water-jacketed. CHEMICAL ABSTRACTS.

Determination of the saturated fraction of fats by means of the thiocyanogen value. H. P. KAUFMANN (Z. angew. Chem., 1928, 41, 1046—1048).—As the thiocyanogen values of oleic and linoleic acids are approximately the same, i.e., 89.93 and 90.57, respectively, the percentage of saturated acids in a fatty acid mixture may be calculated by multiplying the thiocyanogen value by 1.108 and subtracting the result from 100. Similarly, in a mixture of glycerides the percentage of saturated compounds is obtained as above but by using the factor 1.158. The results agree closely with those obtained by the lead salt method and by Bertram's oxidation method. A. R. POWELL.

Detection of rancid edible fats which have been reworked. J. GROSSFELD (Z. Unters. Lebensm., 1928, 55, 376—385).—The octoic acid value of fats (see below) is progressively increased by heating the

fat at 150° or above in contact with air, by treatment with steam, or by development of rancidity, but it is not increased by heating the fat in a current of carbon dioxide or by the process of hardening. Further, the prolonged action of ultra-violet light on normal lard destroys the fraction causing luminescence, raises the octoic acid value, and forms the substance responsible for the Kreis reaction of rancid fats. It is suggested that the increase in the octoic acid value is due to pelargonic and azelaic acids formed by oxidation and fission of oleic acid, and that the substance causing luminescence is a derivative of oleic acid, probably of hydroxyoleic acid. W. J. BOYN.

Octoic acid value for determination of coconut oil. J. GROSSFELD (Z. Unters. Lebensm., 1928, 55, 354—375).—A new octoic acid value for the determination of coconut oil in fat mixtures is defined as the quantity of fatty acid in 0.5 g. of fat which under the given conditions is not precipitated by magnesium sulphate but is precipitated by buffered copper sulphate solution expressed in c.c. of 0.01*N*-solution. A sample of the fat (0.50—0.58 g.) is saponified by warming with 0.2 c.c. of 50% potash and 1 c.c. of glycerin (*d* 1.23). The fatty acids having insoluble magnesium soaps are precipitated by magnesium sulphate solution and collected on a filter next day. From a portion of the filtrate the octoic acid is precipitated by means of a solution containing 50 g. of crystallised sodium acetate, 3.12 g. of crystallised copper sulphate, and 50 c.c. of 20% acetic acid per litre. The precipitate is removed by filtration in a tared Gooch crucible and washed first with a saturated solution of copper octoate and then with 1—2 c.c. of water. Crucible and precipitate are dried at 100° and weighed when cool. The weight of the precipitate in mg. multiplied by the appropriate factor gives the octoic acid value. When less than 2 mg. of copper octoate are weighed the result should be checked by dissolving the precipitate in 20% acetic acid, adding potassium iodide, and titrating the liberated iodine by means of 0.0025*N*-sodium thiosulphate. After deducting the correction obtained from a blank test the octoic acid value is obtained by multiplying the number of c.c. of thiosulphate solution used by the appropriate factor. This method gives results practically identical with those obtained by precipitation of the octoic acid as the silver salt (cf. Bertram, Bos, and Verhagen's method; B., 1926, 499), and avoids the disturbing effect of reducing substances in the soap solution. The octoic acid value found for various fats and for mixtures of coconut oil with butter fat and with cacao fat are given.

W. J. BOYN.

Approximate determination of butter fat and coconut oil in fat mixtures. T. SUNDBERG (Z. Unters. Lebensm., 1928, 55, 397—404).—A diagram has been prepared by plotting the Polenske and Reichert-Meissl values of lard-coconut oil-butter fat mixtures between rectangular co-ordinate axes, and lines have been drawn representing mixtures of equal content of coconut oil and butter fat, respectively. The area so marked out is triangular with two sides straight and the third curved and indicates mixtures containing no butter fat. From the diagram, providing the Reichert-Meissl and Polenske values of the mixture are known,

the contents of coconut oil and butter fat can be read, and also the Reichert-Meissl value due to the butter fat present. This corrected Reichert-Meissl value may be used to obtain, from tables provided, the butter fat content of the mixture when the Reichert-Meissl value of the butter fat is 27 or 29 instead of the value 27.8 on which the diagram is based. W. J. BOYD.

Tung oil. II. D. HOLDE, W. BLEYBERG, and M. A. AZIZ (Farben-Ztg., 1928, 33, 3141—3144; cf. B., 1928, 612).—The iodine value (Hanus) obtained for elæostearic acid is influenced by the time of reaction (*loc. cit.*), the presence of light, and the mode of preparation of the iodine-bromine reagent. Attempts were made to determine the extent of halogen substitution or secondary dehalogenation by determining the halogen acids in the reaction mixture after titration with thiosulphate. Only one fourth to one fifth of the iodine apparently absorbed could be accounted for by the halogen retained by elæostearic acid; the remainder was recoverable from the aqueous solution and appeared to be due, at least in part, to a secondary elimination of halogen acid. It could not be ascertained at what stage in the Hanus test this separation occurred, but it is suggested that there is a tendency for the 6-membered chain constituting the triple conjugated linkings to close, by the addition of halogen to the terminal carbon atoms and subsequent elimination of halogen hydride, leaving hydroaromatic or aromatic derivatives. E. LEWKOWITSCH.

Tung oil. III. Constitution of elæostearic acid. A. EIBNER and E. ROSSMANN (Chem. Umschau, 1928, 35, 197—199; cf. B., 1924, 477; 1926, 448).—By treating the ozonides of α - and β -elæostearic acids with cold water, in the presence of sulphurous acid to avoid oxidation, the authors succeeded in identifying glyoxal (60% yield; isolated as the nitrophenyllosazone) as a product of the reaction. This fact constitutes experimental support for the Böseken-Ravenswaay formula for elæostearic acid (cf. B., 1925, 813). The remaining fragments of the elæostearic acid chain—azelaic and valeric acids and their aldehydes—were identified, but no trace of succinic aldehydes was found. When the ozonide was hydrolysed in the hot, carbon dioxide was freely evolved, but only traces of glyoxal were apparent. It is considered that the small quantities (< 9%) of succinic acid found by von Schapring and by Verduyze among the products of oxidation of elæostearic ozonide by permanganate were due to secondary reactions; tests show that, under such conditions, azelaic acid yields small quantities of succinic acid. E. LEWKOWITSCH.

Constitution of tetradecenoic acid from tsuzu oil. M. TSUJIMOTO (Chem. Umschau, 1928, 35, 225—227).—The crystalline tetradecenoic acid isolated from tsuzu oil (B., 1926, 637) is identified as Δ^3 -tetradecenoic acid by investigation of the ozonide, and is termed "tsuzuic acid." E. LEWKOWITSCH.

Tetradecenoic acid from sperm oil. M. TSUJIMOTO (Chem. Umschau, 1928, 35, 227).—The name "physeteric acid" is given to the Δ^4 -tetradecenoic acid isolated from sperm oil (B., 1925, 856; 1926, 637), the term "physetoleic" acid being already assigned to Δ^8 -hexadecenoic acid found in whale oils. Physeteric

acid is isomeric with tsuzuic acid (cf. preceding abstract). E. LEWKOWITSCH.

Evaluation of oil seeds. P. LIPTÁK (Ber. ungar. pharm. Ges., 1926, 2, 155—158; Chem. Zentr., 1928, i, 1562).—The acid value is determined directly on the alcoholic extract of the ground oil seeds.

A. A. ELDRIDGE.

Extraction of castor oil from the seed with gasoline. A. SLASHCHEV (Masloboino-Zhir. Delo, 1928, No. 3, 9—11).—Benzene darkens the oil; the loss when gasoline is used is 1.5—2%. The use of cold gasoline is unsatisfactory. The characters of standard, extracted, and pressed oils are compared.

CHEMICAL ABSTRACTS.

Purification of vegetable oils. A. ZINOVIEV (Masloboino-Zhir. Delo, 1928, No. 3, 11—15).—To remove slime, crude vegetable oils were heated at 80—100° for 20—30 min., and a current of dry air was blown through them. Similar results were obtained with carbon dioxide, hydrogen, and nitrogen.

CHEMICAL ABSTRACTS.

Sp. gr. of linseed oil. F. FRITZ (Farben-Ztg., 1928, 34, 24—28).—An exhaustive review of the published values for the sp. gr. (and temperature coefficient) of linseed oil. E. LEWKOWITSCH.

See also A., Oct., 1926, **Solubility of alkali soaps in hydrocarbons** (WEICHERTZ). 1092, **Oil emulsions** (KÖHLER). 1103, **Oxidation of fats** (PALIT and DHAR). 1115, **isoOleic acids** (BAUER and MITSOTAKIS). **Esters from Philippine lumbang oil** (VICENTE and WEST).

Sampling pipette. ALLEN.—See I. **Bromometric examination of fats etc.** ANDRISKA.—See XX.

PATENTS.

Manufacture of cream or like fatty powders. J. E. NYROP (B.P. 297,256, 14.12.27).—Fatty material, e.g., cream, emulsions of fats in skim milk, etc., is homogenised and dried by spraying with a "Niro" atomiser (cf. B.P. 259,922) into an atmosphere free or practically free from oxygen and at a temperature sufficiently low to prevent destruction of the vitamins. L. A. COLES.

Manufacture of Turkey-red oils etc. ERBA A.-G. (B.P. 294,621, 15.12.27. Ger., 28.7.27).—Colourless oils are prepared by sulphonating castor oil etc. in the presence of quantities of oxidising bleaching agents, e.g., peroxides, per-acids, and per-salts, in excess of those required for catalytically influencing the sulphonation of the oil (cf. B.P. 292,574; B., 1928, 792). The sulphonation may be preceded and/or followed by treatment with ordinary bleaching agents, fuller's earth, etc. L. A. COLES.

Manufacture of highly sulphonated oils, fats, fatty acids, and waxes. ERBA A.-G. (B.P. 296,935, 30.12.27. Ger., 22.11.27. Addn. to B.P. 294,621, preceding).—Highly sulphonated colourless products are obtained from animal and vegetable oils and fats, fatty acids, or waxes (other than castor oil) by sulphonation with sulphurous acid, or with sulphuric acid or organic sulphonic acids in the presence of oxidising bleaching agents in excess of the amount required to catalyse the sulphonation. After-bleaching may be

performed, if required, and physically acting bleaching agents (e.g., fuller's earth) can be employed.

E. LEWKOWITSCH.

Apparatus for the separation of vegetable oils, e.g., crude olive oil, from juice. AKTIEBOLAGET SEPARATOR (F.P. 620,601, 25.8.26. Spain, 27.8.25).—The oil and juice, after preliminary separation by gravity in a tank, are run through separate inlet tubes into a centrifugal apparatus, the inlet tube for the oil being movable so that it always feeds the oil near the surface, and that for the juice discharging near the bottom of the apparatus.

L. A. COLES.

Oil composition and retardation of development of rancidity therein. W. D. BALDSIEFEN and T. H. ROGERS, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,675,631, 3.7.28. Appl., 9.3.22).—To retard the development of rancidity in castor oil and other oils used as softeners for cellulose nitrate, diphenylamine (0.25–4.0% on the oil) is added.

C. HOLLINS.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Lacquer diluents. A. NOLL (Farben-Ztg., 1928, 33, 3260–3265).—The analysis of various mixtures of aliphatic and aromatic hydrocarbons and alcoholic solvents is described. Qualitatively, ethyl alcohol is detected by formation of its benzoate, benzene by formation of nitro-compounds and colour reactions, and petroleum hydrocarbons by insolubility on nitration or sulphonation. Examination of a series of experimental mixtures demonstrates that "total benzol" content can be obtained with sufficient accuracy for the present purpose either by nitration or sulphonation. In the complete scheme of analysis propounded, alcoholic constituents are determined by absorption by calcium chloride solution, the alcohol in the aqueous layer being obtained, if desired, by distillation methods, and hence the water by difference. The hydrocarbon residue is then nitrated or sulphonated, giving the aromatic content directly and the aliphatic by difference. Typical test reports are quoted, the results of which are to be considered as complementary to such tests as rate of evaporation, distillation range, etc.

S. S. WOOLF.

See also A., Oct., 1920, **Particle size in precipitated zinc sulphide** (LEVI and FONTANA). 1100, **Principles of polymerisation** (SCHEIBER). 1105, **Prussian-blue** (DAVIDSON and WELO).

Tanning agents. BLANC.—See XV.

PATENTS.

Production of high-grade plasters and mixed lacquers containing the same. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,796, 9.4.27).—The products comprise cellulose esters or ethers mixed with at least three times their weight of powdered talc or graphite, or of mixtures of these with lithopone, white lead, ground slate, asbestos, etc., with the addition, if desired, of solvents for the cellulose compounds, plasticising agents, lacquers, resins, etc.

L. A. COLES.

Manufacture of phenolic resins. BRIT. DYESTUFFS CORP., LTD., N. STRAFFORD, and E. E. WALKER (B.P. 296,514, 7.7. and 10.11.27).—Phenol-aldehyde resoles

are incorporated with an inert filler (wood meal, cotton waste, asbestos, china clay), and free phenols and soluble condensation products are extracted with a solvent (water, benzene). When water is used it is advantageous to adjust the p_H to about 6 before extraction. The purified "resole" may subsequently be removed from the filler for use in varnishes, or may be filtered off together with the filler and used as moulding powder. The process is also suitable for production of laminæ, the "filler" being paper sheets.

C. HOLLINS.

Manufacture of flexible abrasive articles such as sandpaper etc. MINNESOTA MINING & MANUF. Co., Asses. of R. P. CARLTON (B.P. 267,516, 8.3.27. U.S., 13.3.26).—Phenol-formaldehyde condensation products in a plastic condition, with or without the addition of solvents or agents for modifying the flexibility, e.g., glycerol, are used as binding agent, and are subsequently hardened by heat.

L. A. COLES.

Manufacture of artificial masses. SOC. CHEM. IND. IN BASLE (B.P. 284,589, 30.1.28. Switz., 29.1.27).—The product obtained by condensing an aromatic amine, preferably aniline, with formaldehyde in the presence of an acid is treated with an alkali to neutralise the acid, after which it is washed, dried, ground to a powder, and compressed, alone or mixed with fillers, coloured material, etc., at the ordinary or at a raised temperature.

L. A. COLES.

Production of material [artificial resin] resembling glass. P. CARPENTIER (F.P. 620,169, 6.8.26).—A solution of casein in 10% sodium carbonate solution is poured into molten phenol, sodium salicylate is dissolved in the mixture, and formaldehyde is then added. After filtration, the mixture is heated under reflux, 80% of the water is removed by distillation *in vacuo*, and the residue is heated at 100° in moulds until polymerisation is complete.

L. A. COLES.

Manufacture of condensation products of urea or its derivatives and formaldehydes. I. G. FARBENIND. A.-G. (B.P. 296,361, 28.2.27. Addn. to B.P. 266,752, B., 1928, 762).—Hydroxymethylcarbamide and other products of mild condensation of formaldehyde with carbamide are treated with alcoholic condensing agents, the treatment being stopped before the formation of high-molecular products. From hydroxymethylcarbamide and methyl hydrogen sulphate in methyl alcohol there is obtained after neutralisation a crystalline product, m.p. 91–92.5°. The product may be further condensed, without elimination of water, to give glassy compounds.

C. HOLLINS.

Manufacture of solvent oil. S. IYORI and K. SUZUKI, Assrs. to ZAIDAN HOJIN RIKAGAKU KENKYUJO (U.S.P. 1,675,977, 3.7.28. Appl., 4.8.23. Japan, 1.11.22).—Resin acids, e.g., colophony or its dry-distillation products, are mixed with fuller's earth, kieselguhr, etc., and distilled at 500°. The main product is an oil, b.p. about 230°, d 0.88, which is a solvent for rubber, pitch, sulphur, etc.

C. HOLLINS.

Production of binding medium [for colours, varnishes, and cements]. J. TENGLER (B.P. 271,073, 9.5.27).—See U.S.P. 1,660,851; B., 1928, 376.

Purification of phenol-formaldehyde resins. F. SEEBACH, ASSR. to BAKELITE GES.M.B.H. (U.S.P. 1,683,702, 11.9.28. Appl., 6.2.26. Ger., 17.2.25).—See G.P. 431,514; B., 1926, 889.

Powdered vitreous polymerised styrene and homologues. I. OSTROMISLENSKY, ASSR. to NAUGATUCK CHEM. CO. (U.S.P. 1,676,281, 10.7.28. Appl., 7.5.24).—See B.P. 233,649; B., 1926, 451.

Method and apparatus for spraying paints, lacquers, etc. for production of a coating upon surfaces. A. GRUBE (B.P. 280,500, 22.7.27. Ger., 15.11.26).

Iron oxide paint (B.P. 296,598). **Red lead** (B.P. 295,975).—See VII. **Moulded articles** (B.P. 282,810).—See IX.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

PATENTS.

Production of rubber goods from vulcanised rubber such as reclaimed rubber, waste rubber, etc. ANODE RUBBER CO., LTD. From P. KLEIN (B.P. 296,107, 23.2.27).—Aqueous dispersions prepared from the vulcanised rubber are caused by electrophoretic, chemical, or mechanical treatment to form deposits upon permanent or temporary backings or moulds. Compounding ingredients may be mixed previously into the dispersions. D. F. TWISS.

Production of aqueous dispersions of organic substances [e.g., rubber etc.]. ANODE RUBBER CO., LTD. From P. KLEIN and A. SZEGVÁRI (B.P. 296,685, 4.3.27).—The organic material, e.g., rubber, reclaimed rubber, or factice, is plasticised and two reagents, e.g., oleic acid and aqueous alkali such as 6% ammonia solution, capable of reacting to form one or more dispersing agents, are introduced while the kneading process is being continued; the aqueous alkali is added gradually and in small amounts. The mass finally becomes fluid, the water then constituting the continuous phase. Compounding ingredients may be incorporated at any stage. D. F. TWISS.

Producing dipped goods from organic dispersions. ANODE RUBBER CO., LTD. (B.P. 285,844, 30.5.27. Ger., 23.2.27).—In the production of shaped goods by repeated dipping operations from aqueous dispersions of organic substances, especially natural or artificial dispersions of rubber, the dipping mould is heated between individual dippings and, if desired, also during the process of immersion. D. F. TWISS.

Production of designs [in colour] on rubber or like materials penetrable by volatile solvents but otherwise non-absorbent. P. ALLMAN and H. N. MORRIS (B.P. 296,461, 3.5.27).—For producing designs on rubber, celluloid, etc. an ink is used comprising a solution of a suitable colour, colour base, or fatty acid salt of the colour in a hydrogenated derivative of phenol, naphthalene, or naphthol, with or without other solvents miscible therewith. D. F. TWISS.

Plastic material. W. B. WESCOTT, ASSR. to RUBBER LATEX RES. CORP. (U.S.P. 1,680,234, 7.8.28. Appl.,

13.1.27).—Asbestos extracted with hydrochloric acid is incorporated with rubber latex. C. HOLLINS.

Preventing separation in liquid dispersions (B.P. 296,454).—See I.

XV.—LEATHER; GLUE.

Depilation of skins by means of alkaline solutions. R. H. MARRIOTT (J. Soc. Leather Trades' Chem., 1928, 12, 342—360; cf. B., 1928, 580, 794).—The hydroxyl ions in fresh lime suspensions hydrolyse the disulphide linkings in the cystine of skin keratins to form calcium sulphide which reduces the remaining keratin more quickly. Clear lime water does not conserve the calcium sulphide formed, and consequently does not loosen the hair on skins. Suspended matter, e.g., lime suspensions, and collagen degradation products, e.g., old lime liquor, retain the sulphides. Since hair which has been treated with hydroxides is less susceptible to the reducing action of sulphides, it follows that fresh lime liquors will not always loosen the hair. The hydroxyl-ion concentration of an old lime liquor is too low to produce this inhibiting effect on the reduction process; the sulphides present from previous use reduce the cystine. There is less dissolved air in an old lime liquor, hence reduction is facilitated. The hair is loosened more quickly by "sharpening" lime liquors with sodium sulphide because of the small sulphide content of old lime liquors. Cystine is probably hydrolysed before it is reduced, and in presence of oxygen the sulphydryl group formed may be oxidised; this will be prevented if reducing agents are present. Most of the ammonia present in lime liquors is derived from the cystine groupings in the hair proteins, and a little is provided by the amides in both hide and skin proteins. The keratins in living or freshly flayed skin contain sulphydryl groups whereas those of dead or preserved skin do not, hence only the former can be un-haired easily in solutions of alkaline hydroxides. D. WOODROFFE.

Biochemistry of soaking [of animal skins]. II. Effect of time, temperature, and hide proportion on soak waters. E. R. THEIS and E. L. McMILLEN (J. Amer. Leather Chem. Assoc., 1928, 23, 372—397; cf. McLaughlin and Rockwell, B., 1925, 682).—Pieces of hide were soaked in four times their weight of water for 1—5 days and the soak liquors analysed. The amount of ammonia present and also the organic, protein, and amino-nitrogen increased as the time of soaking increased, but the amino-nitrogen increased more rapidly than the ammonia, thus showing that the formation of ammonia is not the first criterion of protein degradation. The melanin nitrogen diminished rapidly as the time of soaking increased, probably owing to the bacterial or enzymic destruction of tryptophan. Amide nitrogen is formed through the deamination of the monoamino-acids and through the decomposition of the diamino-acids, and it is shown that these acids decrease during the soaking. The arginine in the hide probably decomposes into urea and ornithine. The former may be oxidised to ammonia and carbon dioxide, the latter to amino-valeric acid or putrescine. The formation of diamines, e.g., putrescine, is a typical reaction of putrefaction and they are easily formed by adding arginine or lysine to a

putrefying medium. The reducing power of the soak increases rapidly after 24 hrs. As the proportion of hide to soak water is increased, the amount of ammonia and organic and protein nitrogen decreases. The optimum hide-water ratio is from 1 : 4—1 : 6, at which proportion less protein degradation occurs. As the temperature rises, a much greater hydrolysis takes place and more protein is dissolved. D. WOODROFFE.

Tanning by means of condensation products of aldehydes and phenols and their derivatives.

C. BLANC (Sealed Note, No. 1928, 9.9.09. Bull. Soc. Ind. Mulhouse, 1928, 94, 359—364). Report by L. MEUNIER (*Ibid.*, 364—367).—Although skins may be tanned by means of phenols and their substitution and oxidation products (quinones), superior results are obtained by use of the condensation products of such substances with formaldehyde. *E.g.*, the product obtained by heating resorcinol with formaldehyde in the presence of a mineral acid is resinous, water-insoluble, and of no value as a tanning agent, whereas a satisfactory tanning agent is obtained by heating for 5—10 min. at 50—60° a mixture of 10 g. of resorcinol, 500 g. of water, 150 g. of sodium bisulphite (d 1.261), and 85 g. of formaldehyde (d 1.083). Skins are tanned by immersion for several hours in the last-named product diluted 3—4 times. Tanning may be effected simultaneously with formation of the tanning agents. Thus, skins may be impregnated and immersed in a suitable mixture of water, resorcinol, sodium bisulphite and formaldehyde, and after a few hours more sodium bisulphite and formaldehyde may be added to complete the condensation and tanning. MEUNIER reports favourably on the process, indicating several related processes, particularly that of Wolesensky (B., 1926, 375). A. J. HALL.

[Acidity of] aqueous extracts from [vegetable-tanned] leathers. E. BÜTTGENBACH (Collegium, 1928, 444—449).—The fat solvent used in degreasing leather does not affect the p_H of the aqueous extract of the leather. Aqueous extracts (Procter extractor) of leather are moderately buffered solutions of a mixture of weak acids of slightly differing dissociation constants. The p_H values of these extracts can be determined to an accuracy of 0.1 by the quinhydrone electrode or by colorimetric methods. The metallic electrode in the former method should be completely surrounded with quinhydrone. The form of the buffering curves supports the use of phenolphthalein as indicator in titrating the acidity of dilute solutions of vegetable tannins and indicates the presence or absence of strong acids in aqueous extracts of vegetable-tanned leathers. Additions of chlorides to these solutions increases their hydrogen-ion concentration. D. WOODROFFE.

Difference of resistance to enzymes of treated and untreated collagen. V. S. SADIKOV (Trans. State Inst. Appl. Chem., Moscow, 1927, No. 6, 82—88).—The resistance of collagen to trypsin is reduced by hydrolysis with water, by rise of temperature, by hydrolase, or by pulverisation. If hides and fascia are pulverised and treated with dilute sodium hydroxide solution their resistance to enzymes is reduced.

CHEMICAL ABSTRACTS.

Preparation of pure collagen and its utilisation

for the determination of tannins in tanning materials. V. S. SADIKOV (Trans. State Inst. Appl. Chem., Moscow, 1927, No. 6, 64—81).—For analytical purposes collagen is preferable to hide powder, which contains also keratin, elastin, proteins, and mucoids. Washed fascia of an ox, after removal of remains of other tissues, is pulverised; the powder is agitated for 6 hrs. (or digested for 24 hrs.) at the ordinary temperature with 0.35% sodium hydroxide solution, the process being repeated twice with fresh solutions. The mass is washed by decantation with water, coagulated with acetic acid or carbon dioxide, pressed, suspended in water, again pressed, and agitated with frequent changes of water. The collagen is finally pressed into thin leaves which are dried, first in cool air (or with acetone or alcohol) then at 40°, and pulverised.

CHEMICAL ABSTRACTS.

Provision of hide powder [for tannin analyses]. TATARSKAJA (Collegium, 1928, 463—464).—Ordinary limed pelt from a tannery is delimed, bated with "Oropon," treated with sodium hydroxide solution for 3 days, washed, brought to p_H 5 with acid, washed, pressed, dried with alcohol, and ground in a specially cooled mill. The product compares favourably with English hide powder in composition and in tannin analyses. D. WOODROFFE.

Effect of acid concentration in plumping on the mechanical and chemical properties of leather. G. G. POVARNIN and SHIMANOVICH (Vestnik, 1926, No. 7, 7—10).—Treatment of calfskin with sulphuric acid before vegetable tanning decreased the tensile strength and resistance to stretch; treatment with lactic acid during tanning had no effect on tensile strength. The "true tanning coefficient" and "true filling coefficient" increased with increased swelling; swelling accompanied by hydrolysis decreases tensile strength and increases roughness. CHEMICAL ABSTRACTS.

Swelling, degree of tannage, and mechanical properties of leather. C. O. POVARNIN and M. LIUBICH (Vestnik, 1925, No. 10—11, 115—118).—Swelling of skin with acids (0.04*N*) decreases the strength; with increasing swelling the "filling coefficient" increases, whilst the "tanning coefficient" becomes maximal and then declines. There is no direct connexion between chemical composition and strength of leather. CHEMICAL ABSTRACTS.

Action of sulphuric acid on leather. A. KUKARKIN (Vestnik, 1925, No. 1, 150—155).—By precipitation of soluble tannin as phlobaphens, the addition of small quantities of sulphuric acid to sole leather increases the apparent percentage of tannage.

CHEMICAL ABSTRACTS.

Resistance of leathers to hydrolysis. G. ARBUSOV (Vestnik, 1926, No. 4, 29—31).—An inconclusive study of the resistance of leathers to hydrolysis before and after extraction with aqueous methyl alcohol.

CHEMICAL ABSTRACTS.

Water-penetration of sole leather. H. VAN DER WAERDEN (Collegium, 1928, 453—458).—The author has used the apparatus previously described by Gerssen (B., 1928, 794) to determine the water penetration of vegetable-tanned sole and dressing leathers, before and

after removing a thin split from grain and flesh sides. There is practically no difference in the water penetrability of these leathers of good quality except that caused by the finishes applied to grain and flesh sides. Quality, filling with extract, or treatment with mineral salt is without effect on this property. Wet leather is more penetrable than dry leather. The water-penetration test is not of much use in judging sole leathers.

D. WOODROFFE.

Rôle of non-tannin. P. I. PAVLOVICH (Vestnik, 1926, No. 5—6, 16—19).—Addition of non-tans to tan liquor is undesirable; addition of acid is preferable.

CHEMICAL ABSTRACTS.

Determination of total acid in tan liquor. M. MICHAILENKO-MALENKO (Vestnik, 1925, No. 9, 52—54).—The liquor is treated with a solution of sodium chloride (50 g.), gelatin (8 g.), and gum arabic (4 g.) in hot water (250 c.c.) neutralised with sodium hydroxide; the mixture is filtered and an aliquot part of the filtrate is treated with 0.01*N*-sodium hydroxide solution, using hæmatin.

CHEMICAL ABSTRACTS.

Colorimetric determination of p_H values in the tannery. L. KÖHLER (Collegium, 1928, 449—453).—A modified Tüpfel method is used. A 1:1 indicator solution and the solution to be tested are mixed on a spotting tile with cavities 0.2 mm. deep. The colour produced is compared with those on a colour chart of the colours obtained at different p_H values. The effect of the natural colour, turbidity, or viscosity is thereby reduced 1000 times as compared with the ordinary colorimetric method. The indicators used are bromophenol-blue, bromocresol-green, methyl-red, bromocresol-purple, bromothymol-blue, phenol-red, cresol-red, and thymol-blue.

D. WOODROFFE.

Comparison of methods of hydrolysis in determining nitrogen in leather. R. W. FREY, L. J. JENKINS, and H. M. JOSLIN (J. Amer. Leather Chem. Assoc., 1928, 23, 397—402).—Four conditions of hydrolysis were compared and the following method is recommended. The leather (1.5 g.) is digested in a Kjeldahl flask with 0.5 g. of copper sulphate crystals, 10 g. of pulverised anhydrous sodium sulphate, and 25 c.c. of concentrated sulphuric acid. After heating below the b.p. until frothing ceases, the solution is boiled briskly so that the condensed vapours of sulphur trioxide hover in the upper half of the bulb of the flask. After 2—2.5 hrs.' boiling, the flask is cooled, the contents are diluted, and the ammonia is distilled off and titrated as usual. Prolonged digestion (4—5 hrs.) may cause loss of nitrogen and is unnecessary.

D. WOODROFFE.

Lecithin reaction for detecting egg yolk in leather. N. JAMBOR (Collegium, 1928, 459—460).—Ordinary finishes are removed from the leather with 1% ammonia solution; nitrocellulose finishes with amyl acetate. The treated leather (10 g.) is then extracted with ether, and to the extract, after removal of the ether by evaporation, are added hot water and 3 c.c. of 10% sodium hydroxide. After boiling the liquor for some minutes, cooling, acidifying with acetic acid, and boiling again it is filtered. Addition of ammonium molybdate in excess to the filtrate acidified with nitric acid produces a yellow precipitate on

warming if lecithin is present. 0.2% of egg yolk used in fat-liquoring was detected.

D. WOODROFFE.

Effect of dilute solutions of gallotannic acid on gelatin. A. KÜNTZEL (Collegium, 1928, 460—463).—The effect on four different grades of gelatin has been determined. The concentrations of the solutions of gallotannic acid at which the gelatin becomes turbid and gives maximum swelling, respectively, are shown to be connected and also to depend on the quality of the gelatin. The differences are attributed to the different isoelectric points of gelatin and gelatose.

D. WOODROFFE.

See also A., Oct., 1092, **Colloidal state of gelatin solutions** (FRANKEL). **Absorption of water by gelatin** (JORDAN-LLOYD and PLEASS).

PATENTS.

Production of soft, tough leather dyed in coloured patterns. N. WAKHROUCHEV (F.P. 618,667, 7.7.26).—Skins are successively softened in water, treated with a solution containing calcium hydroxide and arsenious oxide or sodium sulphide, rubbed to remove the grain, subjected to white- and chrome-tanning processes, and dyed in the usual manner. Portions of the skins are then forced through openings in perforated plates, and the exposed portions only are dyed by treating them successively with solutions containing tannin and a basic dye.

L. A. COLES.

Manufacture of albumin-containing plastic [horn-like] masses having elastic properties. O. MANFRED (B.P. 231,223, 7.6.27. Czechoslov., 26.11.26).—Mixtures for manufacturing the products comprise albuminous material, an aldehyde-ammonia or a condensation product of an aldehyde with an amine or a thioamine, rubber latex, and accelerators and vulcanisers, or, alternatively, powdered caoutchouc or butadiene hydrocarbons, catalysts, and, if desired, protective colloids and fillers.

L. A. COLES.

Manufacture of glue in small pieces, e.g., granules, drops. STOCKHOLMS BENJÖLSFABRIKS AKTIEBOLAG (B.P. 284,704, 30.1.28. Swed., 4.2.27).

XVI.—AGRICULTURE.

Influence of soil reaction on flax and oats. M. DOMONTOVICH and G. ABOLINA (Nauch. Agron. Zhur., 1927, 4, 346—354).—The yield is maximal at p_H 4.1 and 8.1 for oats and p_H 6 for flax. With oats, neutral and alkaline soils produced the largest percentage of nitrogen.

CHEMICAL ABSTRACTS.

Effect of the constituents of alkali fertilisers and soil amendments on the permeability of certain fine-textured soils under irrigation. C. W. BOTKIN (New Mexico Agric. Exp. Sta. Bull., 1927, No. 160, 61 pp.).—Sodium (and potassium) compounds retarded the penetration of water in a clay and a clay-loam soil, the effect decreasing in the order: carbonate, acetate, chloride, nitrate, silicate, sulphate. The following substances (0.5%) increased the rate in decreasing order: aluminium sulphate, ferrous sulphate, aluminium chloride, (primary) calcium hydrogen phosphate, ferric sulphate, potassium hydrogen sulphate, tannin, stable manure, ammonium sulphate. Depressants rendered the soil

more alkaline, and highly deflocculated, and *vice versa*. Fertilisers which tend to decrease soil alkalinity improve permeability and tilth. CHEMICAL ABSTRACTS.

Availability [in soils] of nitrogen in sodium nitrate, ammonium sulphate, and dried blood with varying ratios of phosphoric acid and potash. J. G. LIPMAN and A. W. BLAIR (New Jersey Agric. Exp. Sta. Ann. Rep., 1926, 521—528).—A record of the fourth year of work. A constant amount of nitrogen from three different sources, singly or combined, is used with single, double, or triple portions of phosphoric acid and single and double portions of potassium. The highest average recoveries were obtained with sodium nitrate. For the combined crops (the fertiliser being applied to the first, and the second being grown to utilise any remaining nitrogen), *e.g.*, rape and sorghum, the double portion of potassium gave lower yields and recoveries than the single. CHEMICAL ABSTRACTS.

Influence of acidity and degree of saturation of soils on the uptake of phosphates and potash. F. W. WACKER (Landw. Jahrb., 1928, 67, 589—628; Bied. Zentr., 1928, 57, 343—345).—In the examination of soil nutrients it is important to differentiate between methods measuring nutrient content (*v.g.*, Neubauer seedling method) and those, such as field trials, measuring the nutrient requirement. In the former type of process, experimental conditions largely influence results. Neubauer's method does not yield true nutrient values on very acid or very alkaline soils. It is, however, effective on clay soils within the ranges p_H 5.5—6.2 and 7.7—8.3 (Dirks' method; *cf.* B., 1927, 55), on sandy soils between p_H 6.0—7.5 and 7.3—8.4, and on moorland soils between p_H 6.6—5.5 and 7.6—8.1, in all cases the double p_H figure being initial and final values in this process. The intake of nutrients by plants begins to decrease in the region of p_H 8.1. Hydrolytic acidity is saturated in clays at p_H 7.5—7.6, in sands at p_H 7.2—8.3, and in moor soils at p_H 7.0—8.1. For optimum growth conditions, the reaction of the soil must be adjusted by liming to meet these conditions. Liming to the extent required by the Daikuhara process is insufficient to produce optimum growth of seedlings. A. G. POLLARD.

Increasing the manurial value of Mahua cake. N. D. VYAS (Agric. Res. Inst., Pusa, Bull. No. 176, 1928, 12 pp.).—The effective manurial activity of Mahua cake is but slowly exerted. To increase the rate of nitrification, it is recommended to compost 100 pts. of finely-ground cake with 25 pts. of soil, 5 pts. of charcoal, and 65—70 pts. of water. Fermentation is allowed to proceed for 3 months and the mass is maintained in a slightly moist condition. A. G. POLLARD.

p_H values of plants and corresponding soils. A. V. BLAGOVESHCHENSKI, N. I. SOSEDOV, and A. G. TOSHCHENKOV (Bull. Univ. Asie Cent., Tashkent, 1926, 9—16).—The p_H values of the soils of the West Ferghana salt desert at 10—20 cm. depth were 7.8 ± 0.003 ; the p_H values of press-juice from leaves of various plants ranged from 3.0 to 8.2.

CHEMICAL ABSTRACTS.

Lime and sodium fluosilicate [in insecticides]. S. MARCOVITCH (J. Econ. Entomol., 1928, 21, 436—

437).—When wet plants are dusted with lime and sodium fluosilicate, calcium fluosilicate, which may cause injury to foliage, is formed. An inert carrier for the fluosilicate is preferred. CHEMICAL ABSTRACTS.

See also A., Oct., 1110, Red soils of Cochin China (AGAFONOV). 1161, Maize grown in sand cultures (SPRAGUE). 1162, Rôle of phosphorus in plants (BARRENSCHEEN and ALBERS). Determination of phosphoric acid requirement of soils (NEMEC). Mineral nutrient content of plant solutions (GILBERT, McLEAN, and ADAMS).

PATENTS.

Fungicide or insecticide. I. G. FARBENIND. A.-G. (G.P. 446,530, 11.2.23).—A solution of copper sulphate and 70% of molasses is treated with sodium hydroxide to alkaline reaction, then with calcium naphtholsulphonate, and evaporated to dryness, preferably *in vacuo*, at a low temperature. The product may be mixed with kieselguhr, clay, or chalk for use as a dusting powder or dissolved in water to give a 0.5% solution for spraying.

A. R. POWELL.

Fungicide for plants and seeds. I. G. FARBENIND. A.-G., Assecs. of BADISCHE ANILIN U. SODA FABR. (F.P. 610,281, 30.1.26. Ger., 9.2.25).—Compounds or salts of iron, copper, or mercury with formic acid or formamide form effective fungicides and bactericides for seeds and also promote germination and growth. This applies especially to iron and copper formates and the compound $(HCO \cdot NH \cdot HgCl)_2 \cdot 3HCl$.

A. R. POWELL.

Dry fungicide for seeds. A.-G. F. ANILIN-FABR. (F.P. 599,308, 10.6.25).—The seeds are mixed with a dry powder which evolves a poisonous gas, *e.g.*, kieselguhr impregnated with metaformaldehyde or with oils or other liquids which evolve chlorine. Organic or inorganic salts of copper or mercury may also be incorporated in the mass.

A. R. POWELL.

Insecticide. I. G. FARBENIND. A.-G. (F.P. 617,784, 18.6.26. Ger., 19.1.26).—Esters of formic acid, especially ethyl and methyl formates, are claimed as insecticides; they are more effective than is carbon disulphide and are non-poisonous.

A. R. POWELL.

XVII.—SUGARS; STARCHES; GUMS.

Nitrogenous constituents of molasses and their bearing on its value. H. CLAASSEN (Z. Ver. deuts. Zucker-Ind., 1928, 371—384).—Average data for German molasses from different factories indicate slightly higher nitrogen contents (referred to total non-sugars) for raw-sugar factory molasses than for molasses from white-sugar factories or refineries; but these differences, amounting to about 5—10% of the total nitrogen, are scarcely significant in view of much wider ones (up to 25—30% of the total nitrogen) between individual raw factory molasses. Rational evaluation of beet molasses for yeast manufacture or foddering purposes should take account of the contents of total, assimilable, and amide nitrogen. Assimilable nitrogen may be determined by two successive yeast fermentations, with addition of sugar after the first, as described previously (B., 1926, 686). The amount thus determined is much greater

than that actually assimilated in methods of aeration yeast manufacture where large quantities of ammonium salts are added, because the yeast assimilates the ammonia in preference to other nitrogenous compounds, although the latter are a better form of nutriment. The total amount of nitrogenous matters in beet molasses is about 8.5 times the total nitrogen content, and represents about half the total non-sugars or 80% of the organic non-sugars as usually calculated. To obtain maximum yields of bakers' yeast, the molasses worts should contain 2—2.5 pts. of assimilable nitrogen per 100 pts. of sugar, although only about half of the former passes into the yeast crop; a more complete utilisation of the assimilable nitrogen is only possible at the expense of the quality of the yeast. Beet molasses usually contains, per 100 pts. of sugar, about 3.4 pts. of total nitrogen and 1.4—1.8 pts. of assimilable nitrogen. The necessary excess of the latter may be provided by addition of an extract of malt germs, which contain about 1.8% of total soluble nitrogen and 1.4% of assimilable nitrogen similar in character to that of molasses. Cane molasses contains about $\frac{2}{3}$ — $\frac{3}{4}$ as much organic non-sugar and $\frac{1}{2}$ — $\frac{2}{3}$ as much total nitrogen as beet molasses, and only a small proportion of the nitrogen is assimilable; its value for yeast manufacture lies therefore almost entirely in the sugar content, and it is much poorer in buffer substances than beet molasses.

J. H. LANE.

Clarification of sugar solutions for polarimetry. V. SÁZAVSKÝ (Z. Zuckerind. Czechoslov., 1928, 52, 669—672).—If 26 g. of beet molasses are defecated with basic lead acetate solution and made up to 100 c.c. the volume of the precipitate causes an error of about +0.26 on a polarimetric reading of 50. Such errors are avoided by the use of Horne's dry basic lead acetate defecant, added after the molasses solution has been made up to volume. A slight error in the opposite sense arises, however, from an increase in volume of the liquid due to the space occupied by the excess of defecant in the dissolved state. Excess of defecant must be used to obtain the necessary degree of decolorisation, and an excess of 1 g. is found to cause an error of —0.09 on a Ventzke reading of 50°. J. H. LANE.

Extraction of sucrose from carobs. G. ONDO (Chim. et Ind., 1928, 20, 207—215).—The average composition of carob (locust) beans, without their seeds, is moisture 9—19.5%, nitrogenous matters 2—7%, fat 0.25—0.6%, sucrose 10—34%, reducing sugar 8—28%, starch and other extractives 24—38%, cellulose 4—12%, ash 2—4%, P_2O_5 0.16—0.24%. The author considers that extraction of the sucrose by the ethyl or methyl alcohol process described previously (cf. B., 1928, 29) is economically feasible on a large scale. J. H. LANE.

Danger of inversion in sugar factory products. O. SPENGLER and F. TÖDT (Z. Ver. deut. Zucker-Ind., 1928, 393—405).—According to the hypothesis of ionic activities, the rate of inversion of sucrose in any solution depends only on the temperature and the activity of the hydrogen ions present; and since this activity determines the measured p_H value (as distinct from that calculated from acid concentration), it should be possible to calculate the rate of inversion

at any temperature in any sugar factory product the p_H value of which has been determined. As a basis of reference for such calculations, two graphs showing the rates of inversion for p_H values from 2 to 8 and temperatures from 30° to 130° have been constructed by the authors from the inversion data of Jackson and Gillis (B., 1920, 634). Using these graphs to calculate the rates of inversion at 80° in a number of beet sugars, massecuites, and molasses for which Saillard has determined the actual rates and p_H values, the authors found that the calculated rates either agreed fairly closely with the actual ones or were higher than these. In most cases the ratio of calculated to actual rate was less than 2:1, but in some it was about 10:1, and in one case 35:1. Rates calculated from the p_H values as indicated might therefore prove useful as representing upper limits for the danger of inversion.

J. H. LANE.

New modification of the Clerget method for determining sucrose. J. SCHLEMMER (Z. Zuckerind. Czechoslov., 1928, 53, 13—23).—Clarification is effected by means of bromine liberated in the sugar solution itself by the interaction of potassium bromide and chloramine-*T*. The conditions of actual inversion are those of the Clerget-Herzfeld method, but both direct and inversion polarimetric readings are taken in presence of acetic acid. Applied to beet molasses, for which the method was worked out, the procedure is as follows:—52 g. of beet molasses are diluted to 200 c.c. with water alone, 50 c.c. are treated with a mixture of 10 c.c. of dilute hydrochloric acid (d 1.092) and 20 c.c. of a solution containing 400 g. of sodium acetate and 50 g. of potassium bromide per litre; 10 c.c. of a 15% aqueous solution of chloramine-*T* (Activin) are added with gentle agitation and the liquid is made up to 100 c.c. and filtered for the direct polarisation. For inversion another 50 c.c. of the original solution are treated with 10 c.c. of the dilute hydrochloric acid and inverted according to the Herzfeld procedure, whereupon 20 c.c. of the acetate-bromide solution are run in at once, and, after cooling, 10 c.c. of the chloramine-*T* solution are added, preferably in three portions. The solution is made up to 100 c.c., filtered after about 20 min., and polarised at 20°. For very dark products 0.25—1 g. of Carboraffin or Norit may be added before filtering. The sucrose is calculated by the usual Herzfeld formula except that the divisor 141.75 is substituted for 142.66.

J. H. LANE.

Determination of sucrose by means of alkaline iodine solution. G. BORRIES (Z. Unters. Lebensm., 1928, 55, 405—415).—The method of Auerbach and Bodländer (B., 1924, 107) for the volumetric determination of dextrose has been applied to the determination of sucrose, by finding the difference in the iodine equivalent of the solution before and after inversion. Corrections due to the reaction of the iodine with the laevulose and with the hydrochloric acid used for inversion have been determined. An excess of 9—10 c.c. of 0.1*N*-iodine must be present. The method is suitable for the determination of sucrose in the presence of invert sugar or in artificial honey.

W. J. BOYD.

Evolution of carbon dioxide in the boiling of thin [beet] juices. I. E. THIELEPAPE and P. MEIER (Z.

Ver. deut. Zucker-Ind., 1928, 385—392).—The carbon dioxide evolved in the boiling of thin juices is not derived entirely from bicarbonates; other decomposition processes occur to some extent. Juices boiled under pressure at 116° evolve during the first hour a definite amount of carbon dioxide, which does not appreciably increase during the second hour, and the amount of sugar decomposed is negligible. At 124°, however, considerable decomposition of sugar occurs and carbon dioxide is evolved as long as boiling is continued. At both temperatures the juices, originally alkaline, become slightly acid in time. J. H. LANE.

Abnormal colour of raw [beet] sugar. K. ŠANDELA and A. RUŽICKA (Z. Zuckerind. Czechoslov., 1928, 52, 661—664).—Juices and raw sugars abnormal in colour were obtained in a number of Bohemian factories working beets from a limited area in which hailstorms had done considerable damage to the foliage of the growing plants. The juices after carbonatation were greenish and liable to loss of alkalinity. The separation of the syrup from the raw sugar in the centrifuge was unusually complete, so that the sugars were high in polarisation (e.g., 96.0—97.65) and low in ash content (e.g., 0.6—1.1%). The sugars were greyish and not adapted to long storage owing to loss of alkalinity. There is no record of any special difficulties in refining them. J. H. LANE.

Accurate determination of dry substance in beet-house syrup. R. J. BROWN, J. E. SHARP, and A. R. NEES (Ind. Eng. Chem., 1928, 20, 945—948).—The method described is designed for extreme precision and not for routine use. It is probably unsuitable for cane products containing invert sugar. The material, mixed with sand and some graphite, in an aluminium dish, is dried at 90° for 72 hrs. or longer in a vacuum oven the residual air of which is displaced by carbon dioxide, and then cooled for 3 days over phosphorus pentoxide before re-weighing. J. H. LANE.

See also A., Oct., 1110, Hydrolysis of sucrose by acids (COLIN and CHAUDIN). 1103, Oxidation of carbohydrates (PALIT and DHAR). 1114, Colour reactions of mannitol (EKKERT). 1117, Alkali fission of pentoses (FISCHLER and BOETTNER). Carbohydrate oxidation (EVANS and others). 1119, Carbohydrates from pine wood (HÄGGLUND and others). 1149, Determination of reducing sugars (SEMIGANOVSKY). 1157, Hydrolysis of starch by salts (E. and R. GLIMM). Hydrolysis of sucrose by enzymes (WEIDENHAGEN). 1158, Fermentation of dextrose and laevulose by yeast (HOPKINS). Fermentation of racemised hexoses (FERNBACH, SCHOEN, and MORI). 1162, Starch in pine-needles (BARGUES). 1163, Gum from *Anagyris foetida* (CONDORELLI and CHINDEMI).

Cuban honey. FIEHE.—See XIX.

PATENTS.

Preserving sugar-beets for production of sugar therefrom. B. J. OWEN and J. C. STEAD (B.P. 295,619, 13.4.27).—Whole beets are dried slowly in the dark by warm air at 27—32° until their moisture content is reduced to 5—10%. They are afterwards dry-cleaned to remove soil, and eventually shredded or disintegrated

for sugar extraction. The drying may be carried out by piling the fresh beets in a tall chamber in the centre of which, extending upwards from the floor, is an air-supply chamber with adjustable shuttered outlets for warm air. The roots may first be piled to a depth of 18 in., successive layers being added later as the lower ones become partially dried, until a height of 15 ft. is reached. The drying process occupies 50—60 days. An alternative method is to pile the fresh beets in a trench with sloping sides and with a grating along the bottom forming the roof of a warm-air duct.

J. H. LANE.

Manufacture of sugar. B. H. VARNAU and T. B. WAYNE (B.P. 294,800, 29.9.27).—The syrups derived from the purging of ordinary granulated sugar massecuites in cane-sugar refineries are made to yield, without reboiling, a further crop of extra-fine, soft, granulated sugar. These syrups, delivered from the centrifuges at 60—72°, and at a density of about 78—80° Brix, may, if necessary, be heated sufficiently to redissolve any false grain, and then cooled rapidly to 35—55° to induce the formation of a regular grain, the product being kept in slow motion in crystallisers for $\frac{1}{2}$ —6 hrs. or longer. The degree of cooling necessary depends on the purity of the syrup, and in cases of low purity a seeding magma may be added to the syrup. The sugar which crystallises out is purged and washed in centrifuges. When dry, it polarises 99.5—100, and is free-running and non-hygroscopic; most of the crystals will pass 35—40-mesh. The syrup separated from it may be re-boiled as usual. Apparatus for carrying-out the process is described. J. H. LANE.

Extraction of sugar from beet. R. G. W. FARNELL (B.P. 294,520, 23.2.27. Cf. B.P. 293,066; B., 1928, 685).—Beet cosettes are dried at 100° or below and under atmospheric pressure, and the pressure is then suddenly reduced to facilitate the breaking down of the structure of the beet. J. H. LANE.

Purification of liquids containing sugar. G. E. VAN NES (B.P. 295,831, 18.8.27).—The cold raw sugar juice is treated with lime to render it weakly alkaline to phenolphthalein, and saturated with carbon dioxide until its reaction is acid to this indicator. After heating to 55°, a further quantity of lime is added so that the solution shows a final alkalinity corresponding to about 700 mg. of CaO per litre, and the liquid is allowed to settle for $\frac{1}{2}$ hr. The clear juice is decanted, and the turbid juice remaining is further carbonated, allowed to settle, and the decanted clear juice and the filtered turbid juice are added to a further batch of raw juice in the stage of the purification process before the first settling operation. F. R. ENNOS.

Bleaching and purification of sugar juice. PETROLEUM PRODUCTS REFINING Co. (F.P. 618,779, 15.1.26. Italy, 15.1.25).—The juice, d 1.263—1.383, is treated with nitrogen peroxide under the influence of an electric current, then with a stream of hydrogen, and, after filtration, with a high-tension electric current.

L. A. COLES.

Removal of albuminous substances from saccharine [sugar] juices, molasses, etc. J. POHLMANN and J. R. F. RASSERS (B.P. 287,119, 15.3.28. Holl.,

15.3.27).—Raw cane or beet juices, syrups, or diluted molasses, in a neutral or slightly acid condition, are treated with tannin, tannin derivatives, methyl alcohol, or picric, phosphotungstic, or phosphomolybdic acids to precipitate nitrogenous matters which impede the crystallisation of sugar. J. H. LANE.

Production of [filtered] syrups. F. B. LOMAX (U.S.P. 1,681,490, 21.8.28. Appl., 3.5.26).—A solvent is fed into the two enclosures, above and below a horizontal filter, and the solute is introduced into the upper enclosure in which is an agitating device. The liquid is withdrawn from the bottom enclosure and circulated through the filter until clear. F. G. CLARKE.

Manufacture of dextrose. E. C. R. MARKS. From CORN PRODUCTS REFINING Co. (B.P. 295,830, 15.8.27).—The acid hydrolysis of starch is carried out in the presence of an absorbent, *e.g.*, animal or vegetable carbon free from fats and heavy metals, fuller's earth, silica gel, etc., and in a glass-enamelled vessel which exerts no catalytic tendency to polymerise the dextrose.

F. R. ENNOS.

Manufacture of starch. CORN PRODUCTS Co., LTD., Assees. of R. O. MCCOY (B.P. 288,546, 30.3.28. U.S. 11.4.27).—After disintegration of maize or other starch-bearing materials with water, the whole is subjected to the usual germ separation, and coarse and fine sloop separations, followed by the tabling and washing of the starch. A portion of the effluent from the starch tables is re-used for the sloop separations, and the remainder, after removal of the gluten, is used for steeping the material, whilst the major part of the effluent from the starch washing is returned to the germ separation.

F. R. ENNOS.

Fibres from sugar cane (U.S.P. 1,681,223).—See V.

XVIII.—FERMENTATION INDUSTRIES.

Determination of alcohol by distillation. K. AMBERGER (Z. Unters. Lebensm., 1928, 55, 447—453).—In order to avoid loss of alcohol in distillation, the still wine to be analysed should be diluted so that the distillate contains less than 30 vol.-% of alcohol. Precautions to be observed in fitting up the apparatus and in carrying out the distillation are described.

W. J. BOYD.

See also A., Oct., 1157, **Hydrolysis of sucrose by enzymes** (WEIDENHAGEN). **Emulsin of 23 years ago** (BRIDEL and DESMAREST). 1158, **Fermentation of dextrose and lævulose by yeast** (HOPKINS). **Fermentation of racemised hexoses** (FERNBACH, SCHOEN, and MORI). **Co-zymase** (VON EULER and MYRBÄCK). 1159, **Co-zymase** (VON EULER and MYRBÄCK; NILSSON). **Mutase** (VON EULER and GRABE). **Nucleic acid in yeast** (ISHIYAMA). **Enzymes of *B. coli communis*** (GREY). 1160, **Female sex hormone in yeast** (GLIMM and WADEHN). 1164, **Citric acid production by fungi** (BERNHAEUER). **Fermenting power of *Aspergillus fumigatus*** (WEHMER).

Nitrogenous constituents of molasses. CLAASSEN.—See XVII.

PATENTS.

Acetification apparatus or vinegar generators. H. FRINGS (B.P. 296,950, 21.2.28).—A spent-air duct,

which runs the whole length of the battery of generators, is fitted with siphon devices adapted for the periodic delivery of the appropriate charge of wash to the generators.

C. RANKEN.

Washing of fabrics (B.P. 276,338).—See V.

XIX.—FOODS.

Determination of moisture in buttermilk powder.

T. VON FEELLENBERG and P. HONEGGER (Z. Unters. Lebensm., 1928, 55, 470—473).—Owing to the volatility of the lactic acid, determination of the moisture in buttermilk powder by drying in the oven or by distillation with xylene gives too high results. An error arises also by the formation of lactone and water by the action of heat on lactic acid. The correct result can be obtained as follows. The total water is distilled off with xylene after the method of Mai and Rheinberger (Z. Unters. Lebensm., 1912, 24, 125) whereby some lactic acid and lactone are also found in the aqueous layer. The lactic acid and lactone in the latter are determined by titration before and after saponification with excess of alkali solution. The water formed by decomposition of lactic acid is calculated from the decrease in acidity of the whole sample used. Then the water (*W*) originally present in the sample is given by the formula, $W = (V - V') - 0.2[A - (B + C)]$, where *V* is the volume of the aqueous layer of the distillate, *V'* the sum of the lactic acid and lactone in the distillate expressed in c.c., *A* the lactic acid (in g.) present before distillation, *B* the lactic acid (in g.) in the undistilled residue, and *C* the lactic acid (in g.) in the distillate. W. J. BOYD.

Detection of margarine in butter. H. P. STADLE (Z. Unters. Lebensm., 1928, 55, 404—405).—For the preliminary examination of several samples the Litterscheid pocket polarisation microscope is useful. Adulteration can thereby be detected by observing the presence of starch grains and also, with practice, the occurrence of fat crystals. In testing butter under the quartz lamp a preliminary test may be carried out on the material as received. Pure butter fluoresces yellow, rarely very faintly, whilst margarine shows a blue luminescence. It is best, however, to use a solution of the sample in ether and to examine the insoluble residue microscopically. Pure butter solution fluoresces not at all or only faintly yellow, whilst margarine solution fluoresces so strongly blue that 15% added to butter can be detected.

W. J. BOYD.

Determination of starch in infants' food. T. VON FEELLENBERG (Z. Unters. Lebensm., 1928, 55, 473—475).—The sample is finely ground and 0.25 g. is weighed into a 35 c.c. test-tube. By centrifuging and decanting, the material is extracted first with ether, and then twice with dilute iodine solution. The precipitated starch is dissolved by boiling with 10 c.c. of saturated calcium chloride solution, and the solution is diluted, centrifuged, and the liquid decanted and filtered through cotton wool. The residue is twice washed with 5 c.c. of calcium chloride solution, and the solution diluted, centrifuged, decanted, and filtered as before. The residue is tested with a drop of iodine in case further washing may be necessary. The filtrate and washings are made up to 100 c.c., and the starch in 20 c.c. of this is precipitated by

excess of iodine (2 c.c. of 0.5*N*-iodine solution are usually sufficient); the mixture is centrifuged and the liquid decanted. The residue is washed twice with 5—10 c.c. of 60% alcohol, the alcohol being removed by centrifuging and decanting. The residue is then shaken with 2 c.c. of water and most of this is boiled off in order to remove the iodine and alcohol. This is repeated with 1 c.c. of water. The concentrated starch solution is treated with 10 c.c. of 0.15*N*-potassium dichromate and 20 c.c. of concentrated sulphuric acid. If the solution becomes pure green in colour a further addition of 1—2 c.c. of potassium dichromate solution and sulphuric acid must be made. After 15 min. the treatment is complete, and the solution is diluted to about 350 c.c. When no more gas bubbles are visible, 0.1 g. of potassium iodide is added and the iodine liberated is titrated with 0.1*N*-sodium thiosulphate. A blank test correction is applied. 1 c.c. of 0.1*N*-solution is equivalent to 0.675 mg. of starch. If the sample contains more than 16% of starch either a smaller quantity of the sample must be used or the quantity of dichromate solution proportionately increased.

W. J. BOYD.

Comparative acidimetric investigation of meat extracts, sauces, etc. P. HIRSCH and J. KIESGEN (Z. Unters. Lebensm., 1928, 55, 415—423).—Molar binding power curves of various meat extracts etc. were prepared by electrometric titration. The values obtained for the range p_H 7.0—11.6 were checked by the tropæolin-O, formol, and alcohol titration methods (cf. Tillmans and Kiesgen, B., 1927, 456). Characteristic curves were obtained for different products, so that it should be possible to distinguish meat extracts, meat peptones, and soup preparations thereby. For this it is only necessary to determine the binding power for two p_H ranges, e.g., *a* for p_H 7.0—4.5 and *b* for p_H 7.0—11.6. The value *b/a* was found to be 5.6 for Rotti's soup cubes, 1.02 for Liebig's extract, and 2.27 for a peptone preparation. After preparation of the solution as described the necessary determinations are: (i) titration to p_H 7.0 with neutral-red or phenol-red, (ii) titration to p_H 11.6 with tropæolin-O or by the alcohol or formol methods, (iii) titration to p_H 4.3 with bromophenol-blue using the same coloriscope as for (i) and (ii) and a citrate buffer mixture for comparison.

W. J. BOYD.

Comparison of saltpetre and sodium nitrite as pickling agents for flesh wares. G. RIESS, R. MEYER, and W. MÜLLER (Z. Unters. Lebensm., 1928, 55, 325—354).—By using sodium nitrite instead of saltpetre for pickling flesh products, the time required is reduced to one half or even one third, because the nitrate does not act until it has changed into nitrite. The mechanism of this change, which becomes appreciable only after three weeks, has not been determined. Nitrite gives better penetration of the salt than saltpetre. An addition of 0.6% of sodium nitrite to the salt, corresponding to 0.05% on the weight of flesh treated, is sufficient. If the pickling is properly carried out the nitrite content of the flesh is not greater and may even be less than with saltpetre. The sodium nitrite in the outer layers of non-smoked nitrite-pickled flesh seldom exceeds 10—20 mg. in 100 g. of flesh, while the inner layer contains 1—2 mg. In the outer layers of smoked and nitrite-pickled flesh only a few mg. were present at most. Nitrite-pickled

flesh wares are at least equal in appearance, odour, taste and keeping qualities to those pickled with saltpetre.

W. J. BOYD.

Bacteriology and chemistry of oysters. A. C. HUNTER and C. W. HARRISON (U.S. Dep. Agric. Tech. Bull., 1928, No. 64, 1—75).—The solids, ash, and sodium chloride contents of oysters, meats, and liquor are recorded. Traces of lead and appreciable quantities of arsenic, copper, and zinc were detected.

CHEMICAL ABSTRACTS.

Cuban honey. J. FIEHE (Z. Unters. Lebensm., 1928, 55, 460—466).—An explanation is given of the finding of de Cardenas and Moreno ("Las Micles de Abeja de Cuba," Min. Agric., Comm., and Lab. Cuba) that some samples of genuine Cuban honey give a positive reaction with Fiehe's reagent (1 g. of resorcinol in 100 c.c. of hydrochloric acid), which thus erroneously indicates the presence of artificially inverted sugar. It is pointed out that whereas these authors noted the slightest orange-yellow or reddish coloration as positive, only a strong, persistent cherry-red should be so regarded. De Cardenas and Moreno themselves show that honey produced in districts free from factories and distilleries gives no reaction. It is concluded that the "positive" reaction of some Cuban honey is due to the bees having access to molasses in sugar factories and rum distilleries. This is also supported by the high sucrose content of the samples examined.

W. J. BOYD.

Vanilla, vanillin, and their mixtures with sugar. J. PRITZKER and R. JUNGKUNZ (Z. Unters. Lebensm., 1928, 55, 424—446).—Methods are described for the analysis of vanilla, synthetic vanillin, and their mixtures with sugar, and analytical data are given for products of various origin and quality. The presence of 0.4—0.7% of a volatile essential oil in some vanillas and its absence in others has been ascertained. Vanillin in vanilla is determined on the aqueous extract by a modification of Hanus' method (B., 1905, 1326). Piperonal is similarly determined in the steam distillate from 20—25 g. of vanilla by extracting it with chloroform after adding alkali. In the gravimetric analysis of synthetic vanillin 0.15 g. is dissolved in 50 c.c. of water at 60° and a solution of 0.3 g. of *m*-nitrobenzhydrazide in 15 c.c. of water is added. After $\frac{1}{2}$ hr. on the water-bath at 60° and 24 hrs. at the ordinary temperature the mixture is filtered and the precipitate washed with cold water, dried for 3 hrs. in the water-oven, and weighed. The factor 0.4829 gives the weight of vanillin. If 0.2 g. of sample is used the result is too high, whilst with 0.1 g. it is too low. A quicker and equally satisfactory method is by titration of 25 c.c. of a solution containing not more than 1% of vanillin with 0.1*N*-alkali using 4 drops of 2% alcoholic thymolphthalein as indicator. 1 c.c. of 0.1*N*-alkali corresponds to 0.0152 g. of vanillin. Methods of distinguishing vanillin, bourbonal, and piperonal are given. The above methods of determining vanillin are applicable to vanillin-sugar mixtures.

W. J. BOYD.

Effect of sterilisation on the antiscorbutic vitamin. E. REMY (Z. Unters. Lebensm., 1928, 55, 385—393).—The effect of sterilisation on the vitamin-C content of cauliflower, green beans, green peas, carrots, spinach, and tomatoes has been studied by means of feeding

experiments on guinea-pigs. The vegetables were boiled in water with salt and sterilised for 1 hr. at 98°. The tomatoes were sliced, covered with salt water, and heated at 80° for 20 min. The animals received 50 g. of basal diet mixed with 70 g. of the sterilised material daily besides sufficient water. The Bezssonoff reaction was positive in all cases, strongest in the tomatoes, medium in the green peas and spinach, weaker in the cauliflower, and very weak in the green beans and carrots. The three last-named failed to prevent avitaminosis, and as regards green beans and carrots much larger daily quantities were insufficient. Tomatoes were most effectual, followed by spinach and green peas in the order named. W. J. BOYD.

Fruit jellies. III. Jelly measurements. L. W. TARR (Univ. Delaware Agric. Exp. Sta. Bull., 1926, No. 142, 1—33).—A description of a jelly-strength tester and of the use of citric, tartaric, and sulphuric acids in the production of optimal jellies. Jelly was produced at the same p_H with each acid. The p_H at which the optimal jelly was formed varied with the acid employed. The strength of a jelly is decreased by addition of sugar and increased, within certain limits, by addition of pectin. CHEMICAL ABSTRACTS.

Microscopical detection of durian and nillu (Salpamisri). C. GRIEBEL (Z. Unters. Lebensm., 1928, 55, 453—459; cf. B., 1928, 544).—Further observations are made on the characteristic appearance and chemical reactions of fragments of durian fruit and nillu bulbs under the microscope. W. J. BOYD.

Digestibility trials on Indian feeding stuffs. III. Punjab hays. P. E. LANDER and P. L. C. DHARMANI (Mem. Dept. Agric. India, 1928, 9, 235—246).

See also A., Oct., 1154, **Proteins of linseed and cottonseed meals** (BETHKE and others). 1158, **Clotting of milk by pepsin** (CLIFFORD). 1161, **Determination of vitamin-A** (SHERMAN). **Colour reactions of substances containing vitamin-D** (SEXTON).

Corrosion of tin cans. LUECK and BLAIR.—See X. **Rancid edible fats.** GROSSFELD.—See XII.

PATENTS.

Extracting, separating, and utilising the starch and protein contents of rice. O. JASCHKE (U.S.P. 1,681,118, 14.8.28. Appl., 4.4.25).—Rice is ground dry to a degree of fineness such that the starch granules are liberated; the powder is macerated for 12 hrs. in 0.25—0.3% sodium hydroxide solution, whereby the gluten and fibre swell out and rise to the surface, leaving a thick suspension from which the starch is recovered by centrifuging and settling. A. R. POWELL.

Preservation of eggs, egg yolks, and egg whites. EGG PATENTS, LTD., and A. MILROY (B.P. 296,462, 25.5.27 and 23.3.28).—After removal of the shells, the whole egg, egg yolk or white, or their mixture is mixed with glycerin and the product (preferably finely-divided) is desiccated without unduly raising the temperature (cf. B.P. 261,194; B., 1927, 92). B. FULLMAN.

Treatment of grain, seeds, fruits, and nuts. N. V. INTERNAT. OXYGENIUM MIJ. "NOVADEL" (B.P. 285,859, 22.2.28. Holl., 23.2.27).—The materials are treated with a solution of sulphur dioxide or a sulphite containing an

excess of acid. The residual bleaching agent is removed by centrifuging and washing the grain etc. with alkali, and finally with water, or by oxidation with hydrogen peroxide, heating, steaming, or treating the material under reduced pressure. F. R. ENNOS.

Kneading, mixing, and like machines [for bread, confectionery, etc.]. BAKER PERKINS, LTD., L. S. HARBER, and J. E. POINTON (B.P. 297,197, 9.8.27).

Fatty powders (B.P. 297,256).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Stability and value of chlorine water. G. MIKÓ (Ber. ungar. pharm. Ges., 1926, 2, 163—170; Chem. Zentr., 1928, 1, 1562).—The directions of the Hungarian Pharmacopœia are criticised. A. A. ELDRIDGE.

Bromometric examination of ointments and fats. O. ANDRISKA (Ber. ungar. pharm. Ges., 1927, 3, 140—143; Chem. Zentr., 1928; I, 1563).—The bromine-iodine values of numerous oils, fats, and ointments of the Hungarian Pharmacopœia (3rd ed.) have been determined. The bromoacetic acid method is preferred to the potassium bromate process. A. A. ELDRIDGE.

Comparative examination of commercial cardamoms. W. OTTE and H. WEISS (Pharm. Zentr., 1928, 69, 613—616).—Seven samples of cardamom fruits of varied origin have been examined. Determinations have been made on powdered samples of the whole fruit, seed, husk, and a mixture of equal parts of the whole fruit and husk of the total mineral matter, water-soluble and insoluble mineral matter, ash insoluble in 10% hydrochloric acid, and of raw fibre. The values for the total mineral matter and raw fibre in the husk were more than double the corresponding values for the seed, and the water-soluble mineral matter was 3—4 times as great. An examination of eight commercial samples of powdered cardamom showed that four were definitely adulterated with powdered husk, but, owing to the numerous varieties of cardamom available commercially, definite proof of adulteration of the remaining four could not be given. In conjunction with microscopic examination, however, the method allows of the detection of gross adulteration with powdered husk. Raw fibre determinations by Huggenberg's method give results 1—3% higher than those obtained by the Weender method (cf. B., 1926, 690). E. H. SHARPLES.

Determination of alcohol in tinctures. K. TÄUFEL and U. PANKOW (Apoth.-Ztg., 1928, 43, 240—241; Chem. Zentr., 1928, I, 1897).—A receiving cylinder for use with the German Pharmacopœial (6th ed.) method is described. A. A. ELDRIDGE.

Determination of isopropyl alcohol in alcoholic preparations. C. STAINIER and A. LAUWAET (J. Pharm. Belg., 1928, 10, 167—169; Chem. Zentr., 1928, I, 1897).—*iso*Propyl alcohol is detected in alcoholic preparations by the production of a yellow precipitate when the distillate is heated with an acid solution of mercuric sulphate. Hydrocarbons react similarly. A. A. ELDRIDGE.

Emetine reaction of ipecacuanha preparations. A. WOLLMANN (Ber. ungar. pharm. Ges., 1927, 3, 352—

356; Chem. Zentr., 1928, I, 1564).—The tincture, or the alkaloids extracted from certain preparations, is treated with concentrated hydrochloric acid and 3% hydrogen peroxide; an orange-yellow colour develops slowly in the cold, or more rapidly on warming. The reaction is not accurately quantitative.

A. A. ELDRIDGE.

Determination of morphine in aqueous solution. H. BAGGESGAARD-RASMUSSEN and S. A. SCHOU (Dansk Tidsskr. Farm., 1928, 2, 233—242).—Morphine dissolves in a mixture of 3 vols. of chloroform and 1 vol. of isopropyl alcohol (1 litre dissolves 7.855 g. of morphine) and can be extracted quantitatively by this solvent from aqueous solutions of its salts which have been rendered slightly alkaline with sodium carbonate or bicarbonate. For its determination in aqueous solutions a sample corresponding to a morphine content of 0.1—0.2 g. and not exceeding 20 c.c. in volume is introduced into a separating funnel together with 5 c.c. of 4% sodium bicarbonate solution. A volume of the organic solvent equal to that of the total aqueous solution is added, the whole well shaken for 1 min., and the chloroform layer separated as quickly as possible in order to prevent any crystallisation of morphine from the supersaturated solution. Three further similar extractions are made, the united extracts are filtered, and the solvent is removed by evaporation. The residue is dissolved in 10 c.c. of 0.1*N*-hydrochloric acid, 15 c.c. of water, 2 drops of methyl-red solution, and 1 drop of methylene-blue solution are added, and the excess acid is titrated with 0.1*N*-sodium borate until the colour changes to green. Glycerol and cochineal do not interfere with the method. Fairly accurate results may be obtained when only small amounts (20 mg.) of morphine are present in the solution. H. F. HARWOOD.

Determination of nicotine and ammonia in tobacco. V. OKUDA (J. Biochem. Japan, 1928, 8, 361—364).—The tobacco (3—6 g.) is mixed with water (50 c.c.) and magnesia and distilled with steam; an aliquot part of the distillate is titrated with standard acid, and the ammonia in another part is determined by formaldehyde titration. Alternatively, the ammonia is determined by liberation of nitrogen from nitrous acid.

CHEMICAL ABSTRACTS.

Reactions of novocaine and its distinction from cocaine, alypin, holocaine, and stovaine. L. EKKERT (Pharm. Zentr., 1928, 69, 616—617).—Novocaine with iodine in alkaline solution forms iodoform; with zinc chloride and with *p*-dimethylaminobenzaldehyde yellow to red colours are formed; with furfuraldehyde-sulphuric acid, furfuraldehyde-phosphoric acid, and alcoholic furfuraldehyde alone violet to red colours are produced. Cocaine, alypin, holocaine, β -eucaine, and stovaine do not give the above colour reactions.

E. H. SHARPLES.

Crimean anise oil. B. N. RUTOVSKI and P. LEONOV (Trav. sci. inst. chim. pharm. Moscow, 1924, 10, 64—68).—The oil (0.61%) had f.p. 4°, d_4^{20} 0.9705, α_D^{20} +12.15°, n_D^{20} 1.5405, acid value 0. Phellandrene and methylchavicol, but not fenchone, were detected.

CHEMICAL ABSTRACTS.

See also A., Oct., 1113, Citronellol and rhodinol (GRIGNARD and DUVRE). 1120, Digitalis glucosides (JACOBS and GUSTUS). 1121, Preparation of his-

tidine (VICKERY and LEAVENWORTH). 1127, Mustard oils (DYSON). 1131, Photo-oxidation of adrenaline. 1132, Di(cyclohexylalkyl)acetic acids. 1136, Active component of paracoto bark (SPÄTH and BRETSCHNEIDER). 1137, Cornin: glucoside from *Cornus florida* (MILLER). Sesamin (BERTRAM, VAN DER STEUR, and WATERMAN). 1138, Methylation of shibuol (KOMATSU, MATSUNAMI, and KURATA). Toad poisons. Chinese drug "Senso" (KOTAKE). Hydrogenation of acyclic terpenes (SABETAY and BLÉGER). Citronellal and rhodinol (VERLEY). 1141, Antiseptic compounds from anilquinoline (BROWNING and others). 1143, Homologues of *dl*-ephedrine (HYDE, BROWNING, and ADAMS). 1144, Alkaloids of lupins (SCHOPF). Relations between lupinine and cinchona alkaloids (SCHOPF). 1145, Colour reactions of atropine etc. (EKKERT). Determination of morphine (DÁVID; SCHOLTZ). Colour reaction for quinine (VAN URK). 1146, Thalleioquinine reaction of quinine salts (MIKÓ). Isomerism of *p*-hydroxyphenylarsinic acid (GILTA). Condensation of 4-chloro-3-nitrophenylarsinic acid with amines (FOURNEAU and FUNKE). 1155, Hydrolysis of alkaloids (MAYEDA). Differentiation of *Solanaceæ* alkaloids (GARCIA). 1160, Spirochæticidal properties of vanadium (LEVADITI, LEPINE, and SCHOEN). Crystalline insulin (DIT VIGNEAUD, GEILING, and EDDY; JENSEN and GEILING). 1163, Alkaloid in seed of *Colchicum autumnale* (LIPTAK).

Castor oil. SLASHCHEV.—See XII.

PATENTS.

Iodine compositions [with anthelmintic properties]. Surgical iodine [compositions]. R. W. JAMES. From MERCK & Co. (B.P. 267,557—8, 12.3.27).—(A) Liquids containing proteins, e.g., skim milk or solutions of egg albumin, gelatin, etc., are treated with a quantity of iodine in excess of that required to react with the proteins. The mixture may be administered direct or the adsorption products may be removed by filtration and made up into pills etc. [Stat. ref.] (B) Mixtures of colloidal iodine suspensions prepared as described in B.P. 293,504 (B., 1928, 670) with carbohydrate syrups, e.g., maize syrup, solutions of sucrose, dextrin, gum arabic, etc., are concentrated to a thick syrup for application by means of swabs, or to dryness for dusting as powder on to wounds. L. A. COLES.

Production of preparations having an anti-rachitic action. W., K., L., W., and F. MERCK [E. MERCK] (B.P. 286,665, 23.2.28. Ger., 8.3.27).—Antirachitic provitamin is converted into the vitamin by light of wave-length greater than that of ultra-violet light in the presence of photochemical sensitizers such as iodine or eosin, too intimate contact with oxygen being avoided. These sensitizers also accelerate the similar action of ultra-violet light. E.g., ergosterol is completely converted into the antirachitic vitamin in 2 hrs. if its 0.1% alcoholic solution, containing eosin (5% on the ergosterol), is exposed to the light of a 60-watt lamp.

B. FULLMAN.

Obtaining germ-gland hormones from vegetable organisms. CHEM. FABR. AUF. ACTIEN (VORM. E. SCHERING) (B.P. 277,302, 19.5.27. Ger., 11.9.26. Addn. to B.P. 271,492; B., 1928, 769).—The process

previously described is applied to cherries, parsley root, and yeast. B. FULLMAN.

Preparation of thymol. RHEINISCHE KAMPFER-FABR. GES.M.B.H. (B.P. 293,753, 2.7.28. Ger., 11.7.27).—*m*-Cresol is heated with propylene at 300–350° and 20–40 atm. for 70 hrs. B. FULLMAN.

Manufacture of crystalline menthol. HOWARDS & SONS, LTD., and J. W. BLAGDEN (B.P. 297,019, 8.6.27).—Menthol of m.p. above 34° is obtained by partially esterifying synthetic menthol, e.g., with phthalic acid, removing unchanged menthol by steam distillation, and subsequently hydrolysing the ester. Alternatively, the menthol may be completely esterified and the ester hydrolysed in two or more stages, the menthol formed in each stage being recovered separately. L. A. COLES.

Hydroxy- ω -aminoacetophenone derivatives [hydroxyphenacylamines and their ethers]. H. LEGERLOTZ (U.S.P. 1,680,055, 7.8.28. Appl., 23.3.27. Austr., 22.3.26).—*o*-, *m*-, or *p*-Hydroxyphenacyl halides or their ethers or esters are treated with aliphatic primary or secondary amines; any *O*-acyl group may be removed during the reaction. The following compounds are described: *p*-hydroxyphenacylmethylamine, m.p. 147–148° (hydrochloride m.p. 239–240°; methyl ether); *p*-hydroxyphenacyldiethylamine, m.p. 177–178° (hydrochloride, m.p. 194°; *O*-benzoate). C. HOLLINS.

Pharmaceutical compound. W. KROPP, Assr. to WINTHROP CHEM. CO., INC. (U.S.P. 1,675,500, 3.7.28. Appl., 17.9.25. Ger., 12.12.24).—Methyl salicylate is heated with β -aminoethyl alcohol at 180° to give *salicyl- β -hydroxyethylamide*, m.p. 119°. The 4-methyl derivative, m.p. 88°, is similarly prepared from methyl *m*-hydroxytoluate. The compounds have antirheumatic and antineuralgic properties. C. HOLLINS.

Manufacture of 5-iodo-2-aminopyridine. SCHERING-KAHLBAUM A.-G. (B.P. 288,571, 21.3.28. Ger., 12.4.27).—2-Aminopyridine is treated alternately with iodine (or iodine chloride) and alkali in quantities such that several (e.g., three) additions of the reagents are required for complete conversion into 5-iodo-2-aminopyridine. C. HOLLINS.

Arylazodiaminopyridines useful as bactericides. Pyridium. Neopyridium. Phenyl [benzene] azo- α -diaminopyridine dihydrochloride. I. OSTROMISLENSKY, Assr. to PYRIDUM CORP. (U.S.P. 1,680,108—1,680,111, 7.8.28. Appl., [A] 24.11.23, [B, C] 26.3.26, [D] 23.9.27).—(A) Non-toxic semi-colloidal bactericides, specific against various cocci, are prepared by coupling 2:6-diaminopyridine with diazotised arylamines, especially aniline and *p*-toluidine. The products may be sulphonated and used as soluble sodium salts, or may be applied as hydrochlorides in solution or dispersion, or in ointments. They are without action on the intestinal flora. (B) Either 1 or 2 mols. of diazo compound may be coupled with 1 mol. of diaminopyridine. "*Pyridium*" is the monohydrochloride of benzeneazo-2:6-diaminopyridine. (C) Diazotised anthranilic acid gives an azo compound stable in the form of its inner ammonium salt, from which by action of sodium hydroxide "*Neopyridium*" disodium salt, $C_6H_4(CO_2Na) \cdot N : N \cdot C_5H_2N(NH_2) : NNa$, is obtained. (D) The crystalline dihydrochlorides of the azo compounds are obtained in presence of more than 6% of hydrochloric acid. C. HOLLINS.

Production of quinoline-4-carboxylic acids. A. HÄUSSLER, Assr. to C. H. BOEHRINGER SOHN CHEM. FABR. (U.S.P. 1,676,862, 10.7.28. Appl., 8.12.26. Ger., 6.2.26).—Optimum yields of atophan, 8-phenyl- β -naphthaquinoline-10-carboxylic acid, and 2-*p*-anisyl-6-methylquinoline-4-carboxylic acid are obtained by Döbner's reaction if 2 mols. of aldehyde-amine (or the mixture) are employed to each mol. of pyruvic acid. C. HOLLINS.

Manufacture of new substituted 5:5'-[di]acylamino-4:4'-dihydroxyarsenobenzenes. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 296,327, 27.5.27).—Chlorinated or alkylated arsinic acids prepared by the method of B.P. 278,789 (B., 1928, 37) are reduced, e.g., by sodium hypophosphite, to arsenobenzenes, which are stable to air and produce no injurious after-effects on the nervous system when taken *per os*. 2:2'- and 3:3'-*Dichloro*-, and 2:2'- and 3:3'-*dimethyl*-derivatives of 5:5'-diacetamido-4:4'-dihydroxyarsenobenzenes are described. 2-*Chloro-5-acetamido-4-hydroxyarsenobenzene* (from 2:4-dichlorobenzenearsinic acid), m.p. 187–189°, and 4-*acetamido-5-hydroxytoluene-2-arsinic acid* (from 5-acetamidotoluene-2-arsinic acid) are prepared by nitration, treatment with alkali, reduction, and acetylation. C. HOLLINS.

Medicinal soap. H. H. VON KORNATZKI (U.S.P. 1,684,336, 11.9.28. Appl., 7.8.26. Ger., 26.8.25).—See B.P. 279,575; B., 1928, 37.

Production of esters of aromatic acids [γ -dialkylaminopropyl aminobenzoates]. R. ADAMS and E. H. VOLWILER, Assrs. to ABBOTT LABORATORIES (U.S.P. 1,676,470, 10.7.28. Appl., 9.3.22. U.K., 27.9.21).—See B.P. 191,122; B., 1923, 330 A.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Utilisation of organic compounds as sensitive photographic substances. A. SEYEWETZ (Chim. et Ind., 1928, 20, 216–220).—The use of organic compounds as sensitive photographic substances is confined mostly to the diazo derivatives. Dyes and their leucobases are unsatisfactory on account of their fugacity, their insensitivity to light even in the presence of catalysts, or the lack of suitable solvents for the removal of the products of insolation. J. W. GLASSETT.

Formation of the photographic latent image. S. E. SHEPPARD (Phot. J., 1928, 60, 397–413).—Evidence in support of the assumption that the primary photochemical process is the liberation and transfer of an electron from a bromide ion in the silver halide lattice to a silver ion, forming a neutral silver atom, is afforded by the inner photoelectric effect given by photoconductance phenomena and by the behaviour of photocells composed of two silver electrodes coated electrolytically with very thin films of silver bromide and immersed in potassium bromide solutions saturated with silver bromide. The form of the photopotential-time curves of such cells, obtained on illumination of one of the electrodes, suggests an immediate liberation of both bromine and electrons, but up to the present the initial negative potential, caused in the first fraction of a second after illumination by the rapidly moving liberated electrons, has not been obtained with wave-

lengths longer than 365 m μ , even with the thinnest layers of silver bromide. The present theories of the nature and function of the sensitising specks and of the suggested mechanisms by which the silver produced by photodecomposition is concentrated about them are discussed.

J. W. GLASSETT.

See also A., Oct., 1103, *New photographic phenomenon* (WEIGERT).

PATENTS.

Production of natural-colour photographic pictures. R. GSCHÖPF (B.P. 279,381, 27.5.27. Austr., 22.10.26).—A baryta paper, impregnated with a colloid solution to prevent spreading, is coated with gelatin, sensitised by brushing first with a solution of an iron salt followed by silver nitrate, and then exposed under the first of three part negatives (obtained in the usual way on panchromatic plates by exposure through red, green, and blue filters) until the iron-silver print-out image is visible. The print is then treated with a solution of potassium thiocyanate containing a copper salt and ammonium oxalate whereby the iron is removed and the silver is converted into a colourless silver-copper compound which acts as a powerful mordant for basic dyes. The whole process is repeated on the same base for the other two negatives, the image being dyed with the appropriate colour at each stage. In place of the potassium thiocyanate solution, potassium iodide containing either iodine or potassium ferricyanide may be used, in which case silver iodide acts as the mordant.

J. W. GLASSETT.

XXII.—EXPLOSIVES; MATCHES.

See also A., Oct., 1085, *Mixtures of picric acid and mononitronaphthalene* (JOVINET). 1099, *Detonation-wave in gaseous mixtures* (PAYMAN). *Striae in explosion waves* (CAMPBELL and FINCH).

PATENTS.

[Perchlorate] explosives. C. G. LUIS (B.P. 296,794, 9.3. and 18.3.27).—The explosive contains a perchlorate and the powdered husk or flesh of the fruit of *Terminalia chebula* with or without a small quantity of agar-agar or a vegetable oil. The perchlorate may be the sodium or ammonium salt alone or a mixture of the latter with sodium or potassium perchlorate. The explosive may contain 36–86% of perchlorate and 9–36% of the powdered husk or flesh.

S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Effect of salt on sludge digestion. W. RUDOLFS (U.S. Pub. Health Rep., 1928, 43, 874–881).—The effect of salt on the rate of decomposition of sewage sludge was slight at 5 g. per litre, but with larger quantities the destruction of volatile matter and total gas production markedly decreased, and the composition of the gas changed. With large quantities of salt practically no methane was formed. Mixtures of sodium chloride and sulphate have a stimulating effect.

CHEMICAL ABSTRACTS.

Effect of certain trade wastes on sludge digestion. W. RUDOLFS (U.S. Pub. Health Rep., 1928, 43, 945–951).—A study of the detrimental effect of laundry waste.

CHEMICAL ABSTRACTS.

Pollution problems in the State of Washington and their solution. H. W. NIGHTINGALE (Trans.

Amer. Fish Soc., 1927, 57, 294–300).—The toxicity of sewage and trade wastes to fish life is discussed.

CHEMICAL ABSTRACTS.

Toxicity experiments with fish in reference to trade waste pollution. D. L. BELDING (Trans. Amer. Fish Soc., 1927, 57, 100–119).—The minimum lethal dose of nitric, hydrochloric, or sulphuric acid for brook trout is 1 in 10⁵; loss of equilibrium and irregular respiration are observed at p_H 4–5. Trout survive immersion in 0.5% acetic acid. The effects of phenol, tannin, alkali hydroxides, calcium hypochlorite, copper sulphate, ferrous sulphate, mercuric chloride, potassium permanganate, and hydrogen sulphide are described.

CHEMICAL ABSTRACTS.

Preparation of o-tolidine reagent for free chlorine [in water]. C. S. BORUFF, S. J. VELLENGA, and R. H. PHELPS (J. Amer. Water Works' Assoc., 1928, 20, 404–405).—Assuming an average laboratory temperature of 23°, 1 g. of o-tolidine may be dissolved in a litre of solution containing 178 c.c. of concentrated hydrochloric acid (35%) without depositing crystals on keeping; at lower temperatures a lower concentration of acid is essential (cf. Roake, B., 1925, 333). C. JEPSON.

[Use of dimethyl-p-phenylenediamine hydrochloride as] indicator for chlorine [in water]. K. ALFTHAN (J. Amer. Water Works' Assoc., 1928, 20, 407–411; cf. Kolthoff, B., 1926, 517).—The compound produces a red coloration with chlorine, which is sensitive to 0.1 p.p.m. The reaction requires a p_H value of 2.6–3.4, and is affected by the presence of iron in quantities exceeding 0.1 p.p.m. The red coloration can be matched by comparison with a dilute solution of methyl-red in acid. The indicator used is a 0.4% solution of the hydrochloride containing 8 c.c. of concentrated hydrochloric acid per 100 c.c. C. JEPSON.

See also A., Oct., 1108, *Apparatus for investigation of air* (BUSS). 1163, *Gaseous constituents of river waters* (BUTCHER, PENTLOW, and WOODLEY).

Service pipes for water. LOGAN and EWING.—See X.

PATENTS.

Artificially heating sludge-digestion chambers. M. PRÜSS (B.P. 297,250, 8.12.27).—The optimum temperature for the production of gas from sewage sludge is about 25°. This temperature may be maintained by heating the fresh sludge before admission and by heat-insulating the digestion tanks above and below ground. If sludge is not available, water may be withdrawn from the digestion tanks and returned at a higher temperature. The loss of heat by the portion of the tank below ground may be minimised by surrounding it with a stream of warm sewage. Fresh sludge of high water content should be densified by a preliminary digestion period, and dense sludge should be diluted with warm digestion-tank liquor before admission to the digestion tank proper.

C. JEPSON.

Filtering material and [water] filtration. R. M. MCKEE (U.S.P. 1,676,151, 3.7.28. Appl., 16.9.24).—Waste water is filtered through the absorptive residues produced by distillation of shale oil.

C. HOLLINS.

Insecticide (G.P. 446,530 and F.P. 617,784).—See XVI.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

NOV. 23, 1928.

I.—GENERAL ; PLANT ; MACHINERY.

By-products of chemical warfare. A. A. FRIES (Ind. Eng. Chem., 1928, 20, 1079—1084).—Subsidiary activities of the U.S.A. chemical warfare service have included the following. The protection of piling against marine borers by diphenylaminechloroarsine in creosote or fuel oil was studied, long-period test pieces having been put down in 1924. The toxicity of sodium fluosilicate and a special calcium arsenate (20% As_2O_3) to the cotton boll weevil has been investigated. These are equal weight for weight to ordinary calcium arsenate. Paints for the protection of ships' bottoms have been studied, and the oxides of copper and mercury are recommended as toxic agents with rosin modified by coal tar or synthetic resins as vehicle. A mixture of cyanogen chloride with hydrogen cyanide has been introduced as a fumigant, the lachrymatory effect of the former rendering the mixture much safer than hydrogen cyanide alone. Masks for protection against this mixture, against ammonia, and against carbon monoxide have been designed. Iodine pentoxide is used as a filling in those of the last type and also as a carbon monoxide detector. C. IRWIN.

Technique of mechanical production of highly disperse solutions of solid substances in the ball-mill. K. BERGL and J. REITSTÖTTER (Kolloid-Z., 1928, 46, 53—55).—The mechanics of the colloid mill are discussed and improvements are suggested.

E. S. HEDGES.

Adsorption from solutions and examination of the adsorptive power of medicinal charcoal. T. SABALITSCHKA and K. OEHLKE (Pharm. Zentr., 1928, 69, 629—639, 645—650).—The partial and total adsorptions of methylene-blue from 0.3% aqueous solution, mercuric chloride from aqueous, 90% alcoholic, and 0.1N-hydrochloric acid solutions, iodine from 0.025N-solution in 50% alcohol, phenol from 0.15% aqueous solution, and nicotine from 0.05N-aqueous solution by purified and unpurified, dried and undried commercial samples of beechwood, lindenwood, sponge, bone, blood, and medicinal charcoals, "Carbovent," kaolin, and aluminium hydroxide have been examined. Adsorption-curves are given and the effects of the removal of impurities from the adsorbents and of drying and sieving are discussed. A modification of the German Pharmacopœia VI method for determining the suitability of charcoals for medicinal purposes is proposed.

E. H. SHARPLES.

Technical sedimentation analysis. III. F. V. VON HAHN and A. F. THÖLCKE (Kolloid-Z., 1928, 46, 44—52; cf. B., 1922, 839 A; 1923, 121 A).—The existing methods of preparation of disperse material for sedi-

mentation analysis are criticised. With tooth pastes dispersoid fractions of all particle sizes between 40μ and molecular dimensions are obtained. A single method for obtaining a paste in its primary particles has not been discovered. In many cases the best way is to make the substance into a paste with a soap solution, and in others by Soxhlet extraction with ether, or by boiling with alcohol followed by suspension with water, or by a combination of these methods. The ease with which tooth paste can be suspended in distilled water is related to the surface activity of the paste.

E. S. HEDGES.

Dehumidification of air. C. S. KEEVIL and W. K. LEWIS (Ind. Eng. Chem., 1928, 20, 1058—1060).—It is possible partially to dry unsaturated air by water-cooling without bringing the whole of the air to its saturation point. This fact is discussed with consideration of the temperature and humidity gradients through the surface film, and it is shown that at low humidity the direction of change in the condition of air in contact with water, as shown in the humidity diagram, is towards the point on the saturation curve corresponding to the surface-water temperature. This conclusion was confirmed by passing unsaturated air through a column the walls of which were wetted with water at a temperature below the dew point of the air. The entrance and exit humidities were plotted against the temperatures. For nearly saturated air the curves no longer intersect the saturation curve—an effect which is attributed to condensation in the surface film. The latent heat of condensation in these cases is liberated at the interface and not in the main body of the air.

C. IRWIN.

Waste-heat drying system. WINDSOR and WESTEN-DICK. **Dorr classifiers for clay.** ANABLE.—See VIII.

PATENTS.

[Tunnel] kiln. H. M. ROBERTSON (U.S.P. 1,683,807, 11.9.28. Appl., 28.1.28).—The kiln is provided with a settling chamber (having a clean-out port at one end) which connects a fuel port with a longitudinal combustion chamber.

H. ROYAL-DAWSON.

Apparatus for removing large-sized particles and aggregates from certain finely-divided powders etc. G. GALLIE and B. D. PORRITT, ASSTS. to RES. ASSOC. OF BRIT. RUBBER & TYRE MANUFRS. (U.S.P. 1,688,307, 16.10.28. Appl., 20.3.26. U.K., 7.10.25).—See B.P. 260,741; B., 1927, 32.

Emulsification apparatus. G. C. HURRELL (U.S.P. 1,685,424, 25.9.28. Appl., 12.11.27. U.K., 13.11.26).—See B.P. 285,159; B., 1928, 320.

[Firing of powdered-fuel] furnaces or combustion chambers. E. COCKIN (B.P. 297,345, 16.3.27).

Burners for pulverised fuel. STURTEVANT ENGINEERING Co., LTD., and F. W. R. WILLIAMS (B.P. 297,545, 25.7.27).

Production of fire-extinguishing foam or foam-producing liquids. EXCELSIOR FEUERLÖSCHGERÄTE A.-G., and H. BURMEISTER (B.P. 276,696, 29.8.27. Ger., 28.8.26).

Process and apparatus for producing foam. R. SCHNABEL, J. JACOBSEN, and EXCELSIOR FEUERLÖSCHGERÄTE A.-G. (B.P. 297,782, 24.5.27).

Absorption refrigerating apparatus. ELECTROLUX, LTD., Assees. of PLATEN-MUNTERS REFRIGERATING SYSTEM AKTIEBOLAG (B.P. 290,207 and 275,188, 30.6.27. Ger., [A] 26.7.26, [B] 31.7.26).

II.—FUEL; GAS; TAR; MINERAL OILS.

Low-temperature carbonisation. [Illingworth plant.] D. MUIR (Gas J., 1928, 184, 30—34).—The Illingworth low-temperature plant at Treforest is described. Each retort consists of 18 H-section cast-iron conductors, each 14 in. wide and 12 ft. long, and is slightly tapered from top to bottom (cf. B., 1926, 228, 428). The capacity of each retort is 30 cwt. The flues are of high-silica fireclay. The temperature in the combustion chambers is maintained at about 700°, falling to 650° in the top section. After carbonisation the coke is dropped into a drum containing wet washed smalls, the drum is rotated, and the contents are then screened. This operation serves to cool the coke, dry the coal, and provide an automatic blend suitable for charging into the retorts. The coke forms a good smokeless fuel suitable for either domestic or commercial purposes. From a coal containing 9.6% of moisture, 35.0% of volatile matter, and 4.6% of ash, 15 cwt. of coke and 17.5 gals. of tar per ton of dry coal were obtained. A balance sheet based on the carbonisation of 220 tons of coal per 24 hrs. for 300 working days per year shows a profit of 6s. 2d. per ton.

A. B. MANNING.

Chemistry of coal. V. Maturing of coal considered from the point of view of its benzene-pressure extraction. W. A. BONE, L. HORTON, and L. J. TEI (Proc. Roy. Soc., 1928, A, 120, 523—545; cf. Bone and Quarendon, B., 1926, 305).—Recent criticisms by Colman (J.S.C.I., 1925, 44, 298), by Fischer (B., 1926, 393, 858), and by Cockram and Wheeler (B., 1927, 401, 802) of the benzene-pressure extraction process are discussed. It can be shown experimentally that, contrary to the opinion of Colman, the substances removed by benzene-pressure extraction were not produced by thermal decomposition, in the sense of any real breakdown of the coal substance, but were pre-existent in it, either as such or more probably in some loose molecular association with the coal complex. A comparison between the extraction methods used by Fischer and by the authors indicates that Fischer's method leads to less complete extraction, especially in regard to fraction IV. It is found that in the case of some bituminous coals, fraction II may contribute to the binding properties as well as fractions III and IV. No case has been found in which fraction I has any influence on the coking propensities, nor has it been

found possible to correlate the two in any way. With the object of discovering how the various typical substances comprised in the benzene-pressure extract from a bituminous coal have originated and been developed, an examination has been made of the Morwell brown coal and a number of coals from the Western Canadian coalfield, including a Saskatchewan brown lignite, a series of four black lignites, and a non-coking, semi-bituminous coal. Both fractions I and II of the benzene extract from bituminous and sub-bituminous coals have their counterparts in corresponding fractions obtained from both brown coals and lignites, but although fractions III and IV could also be traced through their counterparts in the laminated black lignites examined, nothing similar to them was found in either the Morwell brown coal or the Saskatchewan brown lignite. In the brown coal and lignite the places of fractions III and IV of the maturer coals were taken by phenolic esters and phenols respectively. It is therefore considered probable that fractions III and IV have originated in the phenols and phenolic esters found in the brown coals. It appears that the general effect of maturing is progressively to diminish the oxygen contents of the substances comprised in the various fractions of the benzene extracts, as well as of the benzene-extracted residue.

L. L. BIRCUMSHAW.

Reactivity of coke. F. J. DENT (Gas J., 1928, 184, 199—200).—Methods for determining the reactivity of cokes in steam, carbon dioxide, and oxygen are described (cf. B., 1926, 970; 1927, 833). Further experiments showed that on blowing air through a column of hot graded coke the highest temperatures are attained by cokes unreactive to carbon dioxide. Ignition-point determinations indicate that the carbonisation temperature is an important factor, high-temperature cokes having high ignition points, and conversely. The chemical properties desirable in cokes for various purposes, viz., for the cupola, blast furnace, producer, water-gas generator, and the domestic grate, are discussed in the light of these experiments.

F. J. DENT.

Coagulation of active charcoal. O. SPENGLER and E. LANDT (Z. Ver. deut. Zucker-Ind., 1928, 549—565).—Suspensions of the charcoal Supranorit 2X are stabilised [peptised] by caustic soda at a concentration of 0.0001N or less, but coagulated at about 0.1N. Coagulation is also brought about by hydrochloric acid and by salts. 1 g. of charcoal shaken with 120 c.c. of liquid is coagulated by the presence of 15 c.c. of 0.1M-sodium chloride, 10 c.c. of 0.1M-ammonium chloride, 10 c.c. of 0.01M-calcium chloride, or 5 c.c. of 0.01M-zinc sulphate. By the use of a polarisation photometer it is possible to follow quantitatively the course of sedimentation. The cations tried exhibit the following relative activity: methylene-blue > H > K > NH₄ > Na > Li; Cu > Pb > Zn; Ba > Sr, Ca > Mg > H; NH₄Et₃ > NH₂Et₂ > NH₃Et > NH₄. The bivalent ions are about 30 times as active as the univalent ions, and the introduction of each ethyl group into the ammonium radical increases the activity about 1.5 times.

F. E. DAY.

Temperatures of welding flames. WEHRMANN (Gas- u. Wasserfach, 1928, 71, 950—952).—The factors

controlling the theoretical flame temperatures of oxy-acetylene and oxy-illuminating gas flames are discussed. The temperature distribution in such flames has been determined by introducing gradually along the axis of the flame cones of refractory material of known m.p., and observing the point at which they melt. Although an oxy-illuminating gas flame develops a slightly lower maximum temperature than an oxy-acetylene flame of the same listed power, the greater efficiency of the latter is not due to this difference, but is due to the greater total quantity of heat produced per unit time.

A. B. MANNING.

Analysis of gas oils and hydrocarbon oils from tars. R. H. GRIFFITH (J.S.C.I., 1928, 47, 21–24 r).—For the determination of different classes of hydrocarbons present in gas oils or tars, a 200 c.c. sample is first distilled from a 250 c.c. flask to give a b.p. curve; 50 c.c. of the distillate are then shaken for 15 min. with 80% sulphuric acid, and the remaining oil, after washing, is distilled to the same end-point. Loss is due to removal of unsaturated substances. Further treatment of the residual oil with 96% sulphuric acid removes aromatic hydrocarbons, and determination of the aniline solution point of this final residue enables the naphthenes to be determined by reference to a standard curve. Figures are also given which show the relation between the aniline solution point of a paraffin hydrocarbon and its b.p. or mol. wt. Examples are included which show the accuracy of the method when applied by independent observers, and demonstrating the results given by a wide range of hydrocarbon oils.

Chemical composition of Rumanian crude [petroleum] oils according to their origin and geological occurrence. N. DANAILA and (Miss) V. STOENESCU (Petroleum, 1928, 24, 1303–1309).—The composition (% of aromatic, olefine, cycloparaffin, and paraffin hydrocarbons) and physical properties are given of 18 Rumanian crude oils and of their fractions, b.p. 65–95°, 95–115°, 115–145°, and 145–300°. Of these oils four come from the Oligocene, ten from the Miocene, and four from the Dacian formation. The composition varies not only from one formation to another (even within the same locality), but also within the same formation. Hence it is impossible to correlate geological origin and chemical composition. The following classification of Rumanian petroleum is based on the content of volatile aromatic hydrocarbons: (1) oil from the Miocene at Moreni or the Dacian at Baicoin, 4% of aromatics; (2) oil from the Dacian at Moreni or Guraoenitei or from the Miocene at Arbanasi, 2½%; (3) from the Oligocene at Solont, Moinesti, Zemes, or Bustenari, or from the Miocene of Bustenari-Gropi or Runcu, 1–1½% of light aromatic hydrocarbons.

W. S. NORRIS.

Artificial ageing of mineral oils. II. F. EVERS and R. SCHMIDT (Wiss. Veröff. Siemens-Konz., 1928, 7, (i), 343–371; cf. B., 1927, 98).—Artificial ageing tests of oils for insulating purposes are carried out by heating the oil at 120° in a closed tube filled with oxygen after previously mixing the oil with a quantity of silica gel impregnated with a catalyst. The catalyst is made by stirring 60 g. of silica gel with 44.48 g. of ferrous sulphate crystals dissolved in water and, after oxidation has taken

place, soaking the mass in a solution of 14.13 g. of ammonium heptamolybdate in 100 c.c. of water, drying, and heating at 410° until ammonia and sulphuric acid are completely expelled. This quantity of catalyst suffices for 15 g. of oil. Curves obtained for American and Russian white oils are of the form $y = c \log x + k$ when the oxygen absorption is plotted against the time; the values of the constants c and k vary with the temperature and with the composition, physical character, and proportion of catalyst used. The oxidation that takes place in the test is practically confined to the constituents with a double linking, e.g., cetene, which become converted into fatty acids with the liberation of hydrogen, carbon monoxide, and carbon dioxide; the presence of unsaturated fatty acids appears to have no influence on the rate of oxidation.

A. R. POWELL.

Determination of the calorific value of Diesel oil. F. V. WARNOCK (Engineering, 1928, 126, 418).—When a bomb calorimeter is not available the calorific value of a fuel oil can be determined within 2–3% by means of a Darling calorimeter. A mixture of the oil (0.75–1.5 g.) with about twice its weight of calcined kieselguhr is loosely packed into a crucible, then covered with a layer of the plain kieselguhr and fired with an iron wire fuse. Heat losses from the instrument may be determined by tests with an oil of known calorific value.

R. H. GRIFFITH.

Pressure of paraffin wax and other oil products at various temperatures and constant volume. L. SELSKI (Azerbeid. Neft. Choz., 1928, No. 4, 69–73).

Adsorption from solutions. SABALITSCHKA and OEHLKE.—See I. **Synthetic hydrocarbons from methane.** FISCHER and others. **Benzene and toluene from xylene etc.** JUCHNOVSKI.—See III. **Solid carbon dioxide.** HOWE.—See VII. **Road surfacing materials.** BUTTERFIELD.—See IX. **Coke-oven gas for Siemens-Martin furnaces.** BULLE.—See X. **Carbon blacks and rubber.** COX and PARK.—See XIV.

PATENTS.

Apparatus for treatment of carbonaceous matter to obtain products therefrom. P. DVORKOVITZ (B.P. 296,793, 8.3.27. Cf. B.P. 192,816; B., 1923, 301 A).—The retorts are arranged in pairs, one above the other, in such a way that after treatment in the upper retort the material can be discharged therefrom into the lower retort and subjected therein to further treatment. Each retort is provided with a rotatable screw or other means of stirring the charge. The retorts are heated externally by the hot combustion products from a furnace, while at the same time a current of preheated neutral gas is passed downward through the material in order to carry off the oils. The first stage of the treatment yields lighter oils, and the second stage heavier oils of the paraffin series.

A. B. MANNING.

Production of gases. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,713 and 296,751, 2.4.27. Addns. to B.P. 214,544; B., 1924, 549).—(A) A producer using granular fuels, as described in the main patent, is provided with a grate composed of two layers of stiffened grate bars arranged in such a way that the slots of the lower sets of bars are masked by the bars of the upper set, the bars being so spaced that neither

the fuel nor the ashes can fall through the grate. The bars slope longitudinally towards clinker ports or pockets. The grate may be of the travelling type. (b) Means are provided for supplying air and steam at a number of different levels in the producer as well as below the grate. This permits complete combustion of the fine dust and some control over the composition of the gas produced. The storage vessel is kept under the same pressure as the air supplied to the producer.

A. B. MANNING.

Purifying gases and gaseous mixtures. J. H. BRÉGEAT (B.P. 296,925, 24.11.27).—The gases are scrubbed with the hydrogenated derivatives of naphthalene, hydrogenated cresols, or hydrogenated terpenes. Naphthalene may be removed from coal gas by scrubbing with tetralin, and may then be recovered in a saleable crystalline form by cooling the solution. The tetralin vaporised by the gas will partly condense in the cooler parts of the distributing pipes, thus aiding in the removal of previously formed deposits of naphthalene; the condensed tetralin containing naphthalene in solution can be collected from siphons attached to the distributing pipes.

A. B. MANNING.

Oxidation of hydrogen sulphide in gas mixtures to sulphur. I. G. FARBERIND. A.-G., Assees. of A. ENGELHARDT and O. MOTSCHMANN (G.P. 447,757, 30.9.25).—In the oxidation of hydrogen sulphide by means of an active carbon or other large-surface catalyst the gases are kept so moist as to be nearly at the dew-point.

C. HOLLINS.

Treatment and fractionation of cracking-plant vapours. E. H. LESLIE and E. M. BAKER (U.S.P. 1,684,771, 18.9.28. Appl., 11.5.23).—Superheated hydrocarbon vapours from a vapour-phase cracking plant are passed up a fractionating column, and a considerable portion of the liquid obtained by condensing the vapour escaping from the top of the column is returned at the top to scrub the ascending vapours.

L. A. COLES.

Apparatus for fractional extraction of petroleum hydrocarbons with alcohol. T. A. WERKENTHIN, Assr. to SOLAR REFINING Co. (U.S.P. 1,680,353, 14.8.28. Appl., 4.11.25).—Petroleum, freed from gasoline and kerosene, is extracted with anhydrous alcohol introduced through pipes at the bottom of the oil tank. The extract overflows through a settler into a still where alcohol is recovered and a cracking oil (35% of the petroleum) is left. The residual oil undissolved by the alcohol is run into a second alcohol-extraction tank where light lubricating oil (10%) is dissolved out. A third (35%) and fourth (10%) extractions give heavier lubricating oils and the residue is asphalt (10%).

C. HOLLINS.

Manufacture of hydrocarbons. I. G. FARBERIND. A.-G. (B.P. 271,452, 9.5.27. Ger., 22.5.26).—The catalytic reduction of the oxides of carbon by hydrogen to produce liquid hydrocarbons and their oxygen derivatives is carried out by passing the mixed gases over catalysts of iron, cobalt, or molybdenum, formed by the decomposition of the corresponding metal carbonyls.

A. B. MANNING.

Manufacture of hydrocarbons. C. ARNOLD. From STANDARD DEVELOPMENT Co. (B.P. 296,536, 8.8.27).—

Finely-divided solid fuel is treated with a hydrocarbon oil and an aqueous liquid in order to separate the bulk of the ash with the latter. The solid fuel moistened with oil is then treated with hydrogen at an elevated temperature and under high pressure, if necessary in the presence of a catalyst. The product is subsequently distilled.

A. B. MANNING.

Cracking of hydrocarbons. F. W. ISOM and C. L. PARMELEE, Assrs. to SINCLAIR REFINING Co. (U.S.P. 1,675,558, 3.7.28. Appl., 3.3.23).—In a cracking process deposition of carbon is minimised by adding a solvent oil to the partly cracked oil so as to keep the tar in solution. A suitable plant is described.

C. HOLLINS.

Apparatus for cracking hydrocarbons. E. C. HERTHEL, Assr. to SINCLAIR REFINING Co. (U.S.P. 1,683,184, 4.9.28. Appl., 11.6.27).—The hydrocarbons are distilled under pressure by circulation through heating coils to and from a horizontal cylindrical drum having a centrally disposed bed of a filtering or absorbent medium. The oil enters the coils from beneath this bed and the cracked oil is discharged into the space above the bed, whence the vapours are also withdrawn for refluxing and condensation.

C. O. HARVEY.

Method of distilling hydrocarbons. F. E. WELLMAN, Assr. to KANSAS CITY GASOLINE Co. (U.S.P. 1,680,580, 14.8.28. Appl., 3.6.22).—Cracking stock is distilled at high cracking temperature and pressure in the first zone, and the vapour produced is fractionated in the second zone, uncondensed vapour from which is conducted off and condensed; the heavier condensed portion is redistilled in a third zone at a temperature lower than that in the first cracking zone, the vapour produced being fractionated in a fourth zone. The light vapour from the fourth zone is removed and condensed but the heavier fraction from this zone is returned to the third zone. All the heavier fractions from the third zone are returned in liquid phase to the first zone.

W. S. NORRIS.

Fractional distillation [of hydrocarbons]. J. W. LEWIS, JUN., Assr. to ATLANTIC REFINING Co. (U.S.P. 1,680,421, 14.8.28. Appl., 25.3.26).—The vapour of the oil is passed counter-current to and in contact with reflux oil, part of which is withdrawn from an intermediate point in the fractionating column and is passed counter-current to and in contact with steam, whereby low components are removed, thus yielding an intermediate fraction of higher flash point; the other part of the reflux oil is continued counter-current to the vapour in an earlier stage of the fractionating system.

W. S. NORRIS.

Cracking and fractionation of hydrocarbon mixtures and apparatus therefor. C. ARNOLD. From STANDARD DEVELOPMENT Co. (B.P. 296,919, 22.10.27).—A stream of a hydrocarbon oil is heated to the cracking temperature, maintained there for a sufficient time to permit any desired degree of cracking, and then passed to a fractionating tower provided with a controlled reflux, in which fractions heavier than kerosene are separated. The uncondensed vapours are passed about midway into another fractionating tower in which the kerosene and gasoline fractions are separated.

A. B. MANNING.

Conversion of hydrocarbons of high b.p. into compounds of lower b.p. I. G. FARBENIND. A.-G. (B.P. 270,313, 28.4.27. Ger., 1.5.26. Addn. to B.P. 268,796; B., 1928, 663).—The following metals may also be employed in the process: titanium, zirconium, niobium, tantalum, thorium, uranium, copper, or thallium, or the metalloids boron and silicon or their alloys. F. G. CROSSE.

Working with hydrocarbons at high temperatures. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,683, 296,693—4, and 296,752—3, Appl., [A—E] 3.3.27).—In order to prevent or to diminish the deposition of carbon or the formation of coke in certain processes in which hydrocarbons are subjected to high temperatures, the surfaces which come into contact with the hydrocarbons at these temperatures are (A) made of a noble metal, ferrosilicon, alloys of chromium, manganese, molybdenum, or cobalt, or steels containing chromium, manganese, or tungsten; (B) made of aluminium; (C) coated with lustrous carbon (cf. K. A. and U. Hofmann, B., 1926, 802); (D) made of metals containing, or having deposited thereon, small quantities of boron, arsenic, antimony, bismuth, phosphorus, selenium or a compound thereof, or silicon; (E) made of molybdenum, tantalum, or chromium. A. B. MANNING.

Producing diolefines from naphtha, naphtha fractions, and naphtha residues. B. W. BYSOW (B.P. 297,231, 20.10.27).—The materials, preheated to 300—400°, are decomposed in a short zone at 750—1000° in the presence or absence of catalysts, and the products are allowed to expand and to be cooled as rapidly as possible, the whole process being conducted under reduced pressure. The gases are subsequently condensed and fractionated. The pyrogenesis may be carried out in the presence of inert gases. W. S. NORRIS.

Treating oil shales and other bituminous materials. W. W. BLAISDELL (U.S.P. 1,684,007, 11.9.28. Appl., 3.5.23).—Before removal of the hydrocarbons by distillation, the material is preheated in a separate chamber by means of the residual gases remaining after condensation of the hydrocarbons. Air is mixed with the residual gases to cool them before they are introduced into the pretreatment chamber, and vapour is not allowed to escape while the material is being transferred from the pretreatment chamber, in which it is agitated, to the distillation chamber. F. G. CLARKE.

Refining or fractionating mineral oils and mineral oil products. A.-G. F. KOHLENSÄURE-IND., and E. B. AUERBACH (B.P. 277,946, 23.8.27. Ger., 25.9.26).—The materials are treated at ordinary temperature in a steel container with liquid carbon dioxide, and the oil-carbon dioxide solution after being separated is freed from the solvent, which is re-liquefied and returned to the process. F. G. CROSSE.

Removal of paraffins from fluid hydrocarbons. AKTIEBOLAGET SEPARATOR-NOBEL and K. G. MALM (B.P. 296,805, 7.6.27).—The paraffins are precipitated, partly at least in the crystalline state, by cooling, and are then separated from the liquid hydrocarbons after dilution of the latter with a solvent which does not dissolve the paraffins at low temperatures. The

centrifugal separator is provided with mechanical means for carrying away the paraffins. Certain products may be first distilled, and fractions thereby separated from which the paraffins are easily recovered by filtration, the residue being then treated as above.

A. B. MANNING.

Compound of kerosene and alcohol. W. HUESSEY (B.P. 267,530, 10.3.27. Ger., 10.3.26).—Motor fuel, for addition to petrol, is prepared by preheating at 160° and 10 atm. a mixture of kerosene and commercial alcohol (containing 5—50% of water), passing the vapours produced through molten lead maintained at 400—500°, and condensing the issuing vapour. The kerosene-alcohol ratio may be, for example, 90:10, and known substances, e.g., bismuth or cadmium, may be used to lower the m.p. of the lead.

W. S. NORRIS.

Lubricants. STANDARD OIL DEVELOPMENT CO., and C. ARNOLD (B.P. 297,068, 9.5.27. Addn. to B.P. 260,602).—In a modification of U.S.P. 1,628,646 (B., 1927, 516) the soap may be formed in the body of the oil by adding the desired base and fatty acid in substantially combining proportions (or by adding the base and an excess of a saponifiable oil, e.g., maize oil) and heating the mixture to 176—190°. When the already formed soap is to be added to the oil, it may be added as an aqueous solution, e.g., a 10% solution. Alternatively, a concentrated solution or gel of soap in oil may be prepared, and this then mixed with more oil in the absence of air (to prevent the formation of an emulsion).

W. S. NORRIS.

Means for preventing sludging in transformer and like oils maintained at high temperatures. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of D. C. COX (B.P. 278,365, 28.9.27. U.S., 28.9.26).—Sludging of the oil in, for example, a transformer is reduced and its useful life prolonged by maintaining a quantity of fuller's earth in contact with the oil. About 6% (by vol.) of fuller's earth is said to increase the useful life of the oil 2—3 times.

W. S. NORRIS.

Method of disposing of acid sludge and spent clay in oil-refining plants. J. M. CORY and F. H. BUNKE, Assrs. to SOLAR REFINING CO. (U.S.P. 1,682,369, 28.8.28. Appl., 27.1.26).—The sludge is agitated with steam, allowed to settle, and the separated acid removed. Spent clay, fuller's earth, coal dust, or other spent oil-purifying material is then added, together with sufficient fuel oil, tank bottoms, or similar oil of low sp. gr. to give a liquid product suitable for use in liquid-fuel burners, and the whole is agitated with steam. The spent purifying material may be added suspended in oil, tar, etc.

F. G. CLARKE.

Distillation of coal and similar carbonaceous substances. C. H. PARKER (U.S.P. 1,687,989—1,687,991, 16.10.28. Appl., 27.1.28. U.K., 25.9.26).—See B.P. 287,584—7; B., 1928, 394.

Stable suspension and paste of coal. R. GRIESSBACH and J. EISELE, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,681,335, 21.8.28. Appl., 23.3.27. Ger., 24.3.26).—See B.P. 292,217; B., 1928, 593.

Producing dry gaseous fuel. E. R. GODWARD. ASSR. to ECLIPSE PETROL ECONOMISER SYSTEM CO., LTD.,

(U.S.P. 1,686,610, 9.10.28. Appl., 23.10.25).—See B.P. 258,222; B., 1927, 930.

Manufacture of fuel [water-]gas. F. WINKLER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,687,118, 9.10.28. Appl., 27.9.23. Ger., 27.9.22).—See G.P. 437,970; B., 1927, 548.

Resolving emulsions of tar or oil. H. W. ROBINSON and D. W. PARKES (U.S.P. 1,687,314, 9.10.28. Appl., 23.2.27. U.K., 30.3.26).—See B.P. 268,547; B., 1927, 469.

Constructive conversion of heavy into light hydrocarbons. W. E. SHORE (U.S.P. 1,687,595, 16.10.28. Appl., 26.12.23).—See B.P. 231,625; B., 1925, 492.

Plant for washing coal and other minerals. A. FRANCE (B.P. 281,605, 26.8.27. Belg., 4.12.26).

Atmospheric gas burners. J. & C. G. BOLINDERS MEKANISKA VERKSTADS AKTIEBOLAG, and E. K. BOLINDER (B.P. 297,647, 26.3.28).

Petroleum-like liquid (Addn. F.P. 31,758). **Phenols and cresols from tar** (G.P. 446,783). **Phenols of coal tar** (G.P., 447, 540).—See III. **Curing cementing materials** (B.P. 291,784).—See IX. **Soaps from mineral oils** (U.S.P. 1,681,237).—See XII. **Plastic material from peat** (U.S.P. 1,681,155).—See XIII.

III.—ORGANIC INTERMEDIATES.

Synthesis of benzene hydrocarbons from methane under ordinary pressures and without catalysts. F. FISCHER [with H. PICHLER, K. MEYER, and H. KOCH] (Brennstoff-Chem., 1928, 9, 309—316).—The yields of tar and light oil formed when methane is passed through a heated tube have been determined. A relatively high temperature (above 1000°) and a high gas velocity are necessary to obtain satisfactory yields of liquid products. The formation of free carbon increases rapidly with rise of temperature, but can be prevented or diminished by correspondingly increasing the gas velocity. Quartz and porcelain are the most suitable materials for the tube; iron and copper favour the separation of carbon. Up to 12.9% of the methane has been converted into light oil and tar by one passage through a heated quartz tube. If the methane is diluted with other gases a higher reaction temperature is necessary in order to bring about the same percentage conversion. The tar contains considerable quantities of naphthalene; the presence of anthracene and phenanthrene has also been established. The light oil consists principally of benzene and toluene. A. B. MANNING.

Preparation of benzene and toluene from technical xylene and from solvent naphtha. G. L. JUCHNOVSKI (Ukraine Chem. J., 1928, 4, [Tech.], 65—87).—Solvent naphtha or technical xylene mixed with hydrogen is passed through metallic tubes heated at 700—800°, and the action of the substance of which the tube is composed, and of various catalysts placed within it, on the yield of benzene or toluene is studied. An unlined iron tube has too intense a catalytic action, less than 54% yields of products boiling below 130° being obtained whilst much soot is deposited. Even lower yields

(43%) are obtained when the tube is packed with iron filings, or with nickel (50%). 64% yields are obtained with iron tubes lined and packed with ferrous sulphide, no soot being deposited, whilst the use of nickel sulphide leads to only 47% yields. Only 43% is given by iron tubes packed with copper turnings; this yield is raised to 56% by lining the tube with ferrous sulphide, whilst replacing it with a copper tube gives a yield of only 2% more. Where sulphide-lined iron tubes packed with alumina catalyst are used, the yields are at first low, owing to oxidation of hydrocarbons, but after using the same catalyst eight times up to 74% yields of products boiling below 135° are obtained, and with this catalyst, which appears to be the best for technical use, no soot formation occurs. The use of magnesia leads to yields of less than 50%. Below 775° no benzene is produced, whilst at 815° the product contains about 43% of benzene. The optimum temperature for this reaction is, however, 775°, as above this naphthalene and gaseous products are formed in increasingly great quantities. The use of coal gas for reduction gives tarry products, difficult to purify, and the same effect is given by substituting nitrogen for hydrogen.

R. TRUSZKOWSKI.

Butyl alcohol. GABRIEL.—See XVIII.

PATENTS.

Conversion of methane into a petroleum-like liquid. A. GOUDET (Addn. 31,758, 9.12.25, to F.P. 613,146; cf. B.P. 255,493; B., 1927, 868).—The fission of the methane into olefines and hydrogen is facilitated by passing the gas at the higher temperatures over a layer of calcium fluoride, to which may be added sulphides, selenides, or tellurides. C. HOLLINS.

Chlorination of acetylene. HOLZVERKOHLUNGS-IND. A.-G. (Austr. P. 106,813, 16.8.23. Addn. to Austr. P. 98,699. Cf. B.P. 174,635; B., 1922, 692 A).—The proportion of chlorine in the prior process is increased to 1.8 vols. to 1 vol. of acetylene with production of dichloroethylene, or to more than 2 vols. with formation of tetrachloroethane. To avoid explosions at least 1—2 vols. of steam must be present for each vol. of chlorine. C. HOLLINS.

Preparation of a catalyst for synthesis of methyl alcohol. SOC. NAT. DE RECHERCHES SUR LE TRAITEMENT DES COMBUSTIBLES (F.P. 610,649, 5.2.26).—An intimate mixture of copper oxide (4 pts.) and zinc oxide (96 pts.) is prepared by precipitation of the mixed salts with boiling alkali; the precipitate is dried and reduced at lowest possible temperature with hydrogen or carbon monoxide. 200—250 g. of methyl alcohol per hr. per dm.³ of this catalyst are produced from hydrogen and carbon monoxide at 150 atm. C. HOLLINS.

Catalytic production of methyl alcohol. COMMERCIAL SOLVENTS CORP., Assees. of J. C. WOODRUFF, G. BLOOMFIELD, and W. J. BANNISTER (B.P. 279,377, 28.3.27. U.S., 25.10.26).—For the hydrogenation of carbon monoxide or dioxide to methyl alcohol a nickel catalyst prepared by reduction at 300—950°, preferably 550°, is used. A gas mixture containing 84% H₂ and 16% CO passed at 20 litres per hr. over 10 c.c. of nickel at 400° under 3000 lb./in.² pressure gave 6 c.c. of 50%

methyl alcohol per hr.; at 3.5 litres per hr. 1.1 c.c. of 82% methyl alcohol were obtained per hr.

C. HOLLINS.

Sulphonation of fatty acids and their esters. H. T. BÖHME A.-G. (B.P. 263,117, 27.11.26. Ger., 16.12.25. Addn. to B.P. 261,385; B., 1928, 418).—By increasing the proportion of acid anhydride (or chloride) used in the sulphonation process of the prior patent more highly sulphonated products are obtained. These have excellent wetting-out and foaming properties even in acid solution, are not salted out by salt or concentrated alkali, and are useful additions to mercerisation liquors. A good product is obtained by sulphonation of a mixture of equal weights of castor oil and acetic anhydride.

C. HOLLINS.

Rendering higher alcohols soluble in aqueous media. H. T. BÖHME A.-G., and H. BERTSCH (B.P. 272,919, 15.6.27. Ger., 15.6.26).—Butyl and higher alcohols give clear solutions in water, alkali, or dilute (carbonising) acid when mixed with the sulphonates prepared according to B.P. 261,385 or 263,117 (B., 1928, 418, and preceding).

C. HOLLINS.

Manufacture of formaldehyde from methylene dichloride. E. KRAUSE and K. RÓKA, Assrs. to HOLZVERKÖHLUNGS-IND. A.-G. (U.S.P. 1,679,673, 7.8.28. Appl., 23.10.24. Austr., 17.9.21).—Methylene dichloride is heated at 130–150° with 5% aqueous ammonia in a closed vessel, the ammonia being added gradually to avoid strong alkaline reaction.

C. HOLLINS.

Manufacture of paraformaldehyde. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,847, 1.7.27).—Further to B.P. 267,768 (B., 1927, 428), the reaction gases from the catalytic oxidation of methyl alcohol vapour are cooled to 50° and washed with 60% formaldehyde solution at 50°. Water and formaldehyde condense, but the wash liquors remain at about the same concentration; excess liquor is run off continuously and on keeping changes to a tallow-like paste of paraformaldehyde.

C. HOLLINS.

Manufacture of monoaryldiguanidine salts of substituted dithiocarbamic acids. I. G. FARBENIND. A.-G., Asses. of E. SÖRENSEN (G.P. 448,631, 24.3.25).—A mixture of a monoaryldiguanidine and a primary or secondary amine is treated with carbon disulphide. Phenyl- and *o*-tolyl-diguanidine phenylethylthiocarbamates and *o*-tolyl-diguanidine phenylthiocarbamate are described. If commercial ethylaniline is used, the diethylaniline present is recovered.

C. HOLLINS.

Manufacture of heavy-metal salts of disubstituted dithiocarbamic acid. I. G. FARBENIND. A.-G., Asses. of E. SÖRENSEN (G.P. 448,632, 14.3.25).—An ammoniacal solution of zinc chloride, copper sulphate, or nickel sulphate is stirred with carbon disulphide and a secondary amine, e.g., methyl- or ethyl-aniline, or ethyl-*o*-toluidine; the technical mixture of secondary and tertiary amines is used with advantage. No cooling is needed, since no heat is evolved. Yields are 75–95%.

C. HOLLINS.

Manufacture of new compounds from indene and phenols [disinfectants; resins for lacquers]. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 297,075, 14.6.27).—Indene (or solvent naphtha, b.p.

160–190°) condenses with phenol in presence of hydrochloric acid to give an oil, b.p. above 180°/9 mm., which on dissolution in cyclohexane deposits 3-*p*-hydroxyphenylhydrindene, m.p. 88° (compound with hexamethylenetetramine, m.p. 112°), a non-toxic disinfectant. Crude cresol gives an oil, b.p. 180–230°/9 mm., and a spirit-soluble resin; *o*-cresol yields 3-(6-hydroxy-*m*-tolyl)hydrindene, m.p. 63°, b.p. 200–230°/10 mm., and a viscous oil. The product from resorcinol distils at 220–260°/2 mm. and sets to a glassy mass. 1-Xylylindene, obtained from indene and crude methylbenzyl chloride, condenses with phenol or cresols to give an alkali-insoluble, viscous oil, b.p. 200–250°/10 mm.

C. HOLLINS.

Manufacture of unsaturated hydrocarbons, esters, and salts [camphene, isobornyl acetate, sodium acetate]. M. ALLINGER (F.P. 583,341, 28.9.23).—Cuprous oxide or a cuprous salt (or a cupric salt and a reducing agent) is used to remove halogen from organic hydrohalides or from inorganic halides, particularly in presence of acetic acid to form acetic esters or inorganic acetates. Pinene hydrochloride heated with cuprous oxide gives camphene, or, in presence of sulphuric and acetic acids, isobornyl acetate. Sodium chloride, cuprous oxide, and acetic acid, stirred for 15 min. with exclusion of air, give sodium acetate.

C. HOLLINS.

Manufacture of highly chlorinated perylenes. A. ZINKE and K. FUNKE, Assrs. to F. BENSA (U.S.P. 1,681,077, 14.8.28. Appl., 7.6.27. Austr., 2.4.27).—7 to 12 atoms of chlorine may be introduced into perylene by chlorination in nitrobenzene suspension in presence of aluminium chloride or antimony pentachloride.

C. HOLLINS.

Treatment of partial oxidation products. J. H. JAMES, Assr. to C. P. BYRNES (U.S.P. 1,681,185, 21.8.28. Appl., 5.11.19).—Acids are separated from aldehydes etc. in oxidised paraffins and converted into salts.

C. HOLLINS.

Purification of partial oxidation products. J. H. JAMES, Assr. to C. P. BYRNES (U.S.P. 1,681,238, 21.8.28. Appl., 14.12.25).—Unsaturated constituents of oxidised liquid hydrocarbons are hydrogenated or otherwise converted into saturated products.

C. HOLLINS.

Conservation of food, provisions, technical and pharmaceutical preparations, etc. and manufacture of disinfecting preparations. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 297,074, 14.6.27).—Phenol ethers containing no free hydroxyl group have greater antiseptic power than the free phenols, are non-corrosive, do not precipitate proteins, and are insensitive to alkali. Examples are veratrol, anisic acid, and acetanisidide. Reference is made to Public Health (Preservatives etc. in Food) Regulations, 1925.

C. HOLLINS.

Manufacture of benzene and its homologues from mixtures of oxides of carbon and hydrogen. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,179, 13.7.27).—Water-gas at 50 atm. is passed at 40 litres per hr. per litre of catalyst over an iron-silver catalyst at 370°, and the resulting mixture of olefines and paraffins, after being cooled in a heat-exchanger and washed at 30 atm. with water, is passed at ordinary

pressure through a copper tube filled with iron-free copper turnings at 800° at a speed of 60 litres per hr. per litre of tube. The gas after the first stage before washing contains 13% of olefines, 10% of ethane, and 4% of methane; in the washed gas these figures are increased to 32%, 25%, and 10%, and this mixture yields an equal weight of 80% benzene in the second stage.

C. HOLLINS.

Recovery of phenols and cresols from crude tar. GELSENKIRCHENER BERGWERKS A.-G., and F. SCHÜTZ (G.P. 446,783, 30.9.23).—Crude tar of b.p. 170–360° is agitated with a dilute solution of sodium hydroxide containing just sufficient alkali to dissolve the phenols with low b.p. The aqueous layer is separated, filtered through sand or coke, and treated with a stream of carbon dioxide on the counter-current principle. The precipitated phenols are then separated by fractional distillation.

A. R. POWELL.

Isolation of *p*-xylene and *m*-4-xylene from phenols of coal tar. GES. F. TEERERWERTUNG M.B.H., Assees. of E. MOEHRLE (G.P. 447,540, 27.2.26).—The tar-xylene fraction, b.p. 207–211°, is sulphonated as usual and the *p*-xylenesulphonic acid which separates on dilution and keeping is hydrolysed to *p*-xylene with concentrated sulphuric acid and superheated steam. The sulphonation mother-liquor is added to a cold saturated solution of potassium chloride, whereby potassium *m*-4-xylenesulphonate is at once precipitated. This re-dissolves on warming and crystallises out as the solution cools. The sulphonate is hydrolysed to give *m*-4-xylene, b.p. 208.5–210°, as an oil which sets readily when seeded. The yields are 15–17% of *p*- and 25–30% of *m*-xylene.

C. HOLLINS.

Manufacture of *p*-methylaminophenol. K. FRICKER (G.P. 449,047, 3.10.23).—*p*-Aminophenol is methylated by boiling with 1 mol. of methyl sodium sulphate in water for 6–8 hrs.

C. HOLLINS.

Production of aromatic amines. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,212, 10.9.27).—Aromatic nitro-compounds are reduced to amines by hydrogen under 140–200 atm. pressure in presence of a metal catalyst (iron powder, spongy iron, nickel deposited on zeolite). Catalyst poisons (*e.g.*, sulphur) must be present in the nitro-compound and/or the reducing gas to diminish the activity of the catalyst. Reaction begins at about 100°; 150–200° is recommended.

C. HOLLINS.

Manufacture of formyl compounds of [amines of] the aromatic series. I. G. FARBENIND. A.-G., Assees. of R. WIETZEL (G.P. 449,112, 12.3.25).—Formanilide (*etc.*) is obtained by the interaction of formamide and aniline (*etc.*) at reduced pressure (*e.g.*, 10–30 mm.) to facilitate removal of ammonia. High temperatures are unnecessary. Reaction is facilitated by presence of water (added continuously up to a total of 10–20%), or formic acid, or by a stream of inert gas. The formanilide produced is technically pure, and may be used directly for manufacture of *p*-nitroaniline.

C. HOLLINS.

Intermediate compounds for preparation of dyes. BRIT. DYESTUFFS CORP., LTD., J. B. PAYMAN, and

E. G. BAINBRIDGE (B.P. 296,458, 2.5.27).—1:8-Naphthasultone-3-sulphonic acid is converted by phosphorus pentachloride at 140° into the 3-sulphonyl chloride, which reacts with dry ammonia to give the 3-sulphonamide, with aniline to form the 3-sulphonanilide or with cold aqueous ammonia to give α -naphthol-3:8-disulphonamide. The 3-sulphonanilide is converted by aqueous sodium hydroxide into 8-sulpho- α -naphthol-3-sulphonanilide or by ammonia into 8-aminosulpho- α -naphthol-3-sulphonanilide.

C. HOLLINS.

Manufacture of amines of the hydroaromatic-aliphatic series. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,484, 23.5.27).—Ethylene oxide reacts with cyclohexylamine in water at 0° to give cyclohexyl- β -aminoethyl alcohol, m.p. 50°, b.p. 123–127°/14 mm., and cyclohexyldi-(β -hydroxyethyl)-amine, b.p. 180–184°/14 mm. The secondary amine is also obtained from cyclohexylamine and ethylene chlorohydrin, or by catalytic hydrogenation, in presence of nickel at 130–140° and 35 atm., of a mixture of cyclohexanone and alcoholic β -aminoethyl alcohol. Similar products are obtained from homologous cyclic ketones and aminoalcohols, or by condensing alicyclic oxides with aminoalcohols. The products are insecticides and bactericides, and their acyl derivatives are plasticisers.

C. HOLLINS.

Purification of naphthalene. COMP. DE PROD. CHIM. & ÉLECTROMÉTALLURGIQUES ALAIS, FROGES, & CAMARGUE (F.P. 602,408, 26.11.24).—Crude naphthalene is heated at 100° with 7% of the chloride of sulphochloroacetic acid (obtained from trichloroethylene and 100% sulphuric acid). Sodium hydroxide is added and an easily hydrogenated, pure naphthalene is distilled off in steam, leaving the impurities as a resinous, non-volatile residue.

C. HOLLINS.

Manufacture of sulphonic acids of aralkylated polynuclear aromatic or hydroaromatic hydrocarbons. I. G. FARBENIND. A.-G., Assees. of K. DAIMLER and G. BALIE (G.P. 449,113, 23.7.24).—Saturated or unsaturated alcohols are condensed with the products from aralkyl halides and polynuclear aromatic or hydroaromatic hydrocarbons, before, during, or after sulphonation, to give compounds with improved frothing, wetting-out, and emulsifying properties for application in the dyeing and textile industries. Examples are: benzylated naphthalene treated with *n*-butyl alcohol and oleum and chlorosulphonic acid at 70–80°; naphthalene- β -sulphonic acid with benzyl chloride, *n*-butyl alcohol, and concentrated sulphuric acid at 100–120°; benzylated naphthalene sulphonated at 60–100° with 20% oleum and chlorosulphonic acid, and the reaction mixture treated with cyclohexanol at 80°; benzylated tetrahydronaphthalene sulphonated and condensed with isobutyl alcohol.

C. HOLLINS.

Manufacture of hydrogenated naphthastyril I. G. FARBENIND. A.-G. (B.P. 281,257, 9.11.27. Ger., 25.11.26).—Naphthastyril is hydrogenated in presence of nickel at 100–150° and 20 atm. to give 10:3:4:5-tetrahydronaphthastyril (*N* = 1), m.p. 125–126°.

C. HOLLINS.

Manufacture of 7-acylamino-1:4-naphthoquinones. I. G. FARBENIND. A.-G. (B.P. 291,340,

29.5.28. Ger., 28.5.27).—7-Acetamido- α -naphthol is oxidised with chromic-acetic acid below 0° to 7-acetamido-1 : 4-naphthaquinone, decomp. 228—230°. The 7-benzamido-compound, decomp. 232°, is similarly prepared. C. HOLLINS.

Manufacture of 4-nitro-2-amino-1-benzoic acid [4-nitroanthranilic acid]. I. G. FARBENIND. A.-G. (B.P. 285,877, 23.2.28. Ger., 24.2.27).—2-Chloro-4-nitrobenzoic acid is heated with 33% aqueous ammonia and copper powder at 120° in a closed vessel to give 4-nitroanthranilic acid, m.p. 279°. C. HOLLINS.

Manufacture of derivatives and condensation products of anthraquinones [benzanthrone]. W. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 297,129, 12.5.27).—Anthrones are condensed in sulphuric acid with cinnamaldehyde, and the products are converted by aluminium chloride or heat into benzanthrone. Cinnamylideneanthrone, heated with aluminium chloride at 100—105°, gives benzanthrone, but when heated alone or with 1-chloronaphthalene, *p*-phenetidine, sodium acetate, phosphoryl chloride, etc. it is converted into 3-phenylbenzanthrone, m.p. 181°, which is also obtainable in smaller yield by heating together cinnamaldehyde, anthrone, and the solvent or condensing agent. Cinnamylidene derivatives of the anthrones from 1-hydroxy- and 2-chloro-anthraquinones similarly yield substituted 3-phenylbenzanthrone. Treatment of 3-phenylbenzanthrone with aluminium chloride causes migration of the phenyl group to the 2-position (2-phenylbenzanthrone, m.p. 199—200°). C. HOLLINS.

Manufacture of new condensation products of the benzanthrone series. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 296,490, 9.6.27).—3-Nitro-4-methylbenzanthrone, m.p. 243°, is converted by 60% oleum, or by a mixture of 70% oleum and chlorosulphonic acid, or by aluminium chloride in trichlorobenzene into grey to black vat dyes, together with the lactam of 3-aminobenzanthrone-4-carboxylic acid, m.p. 320°, which may be removed by extraction with acetic acid, and yields on hydrolysis the 3-amino-4-carboxylic acid, m.p. 278°. C. HOLLINS.

Manufacture of anthrahydroquinone [dihydroanthraquinone] derivatives. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 297,042, 13.4.27).—2-Acylaminoanthraquinones are reduced by hydrogen at 20 atm. in presence of nickel in a solvent (pyridine, chlorobenzene) or in alcoholic suspension, or in aqueous alkali to give 2-acylamino-*ms*-dihydroxyanthracenes, which are converted by the usual methods into ethers or esters, with or without removal of the acyl group. 2-Acetamido-9 : 10-dihydroxyanthracene (dimethyl ether, m.p. 253°) gives with pyridine and chlorosulphonic acid the dihydrogen disulphate, from which the acetyl group may be removed by boiling with alkali; the dihydroxy-compound condenses with chloroacetic acid to give the glycollic acid [di(carboxymethyl) ether], m.p. 240°, from which the acetyl group may similarly be removed. C. HOLLINS.

Production of dye intermediates [pyrazolanthrone]. I. B. ANDERSON, R. F. THOMSON, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 297,366, 18.3. and

25.6.27).—An α -chloroanthraquinone is condensed with hydrazine sulphate in presence of calcium oxide or hydroxide, sodium carbonate, or sodium acetate at 180—200° in a closed or open vessel. With small amounts of the alkaline agent (3 mols.) an anthraquinonylhydrazine is formed, but with excess the product is the pyrazolanthrone. C. HOLLINS.

Manufacture of acetaldehyde. H. S. REID and W. C. HOVEY, Assrs. to CANADIAN ELECTRO PRODUCTS CO., LTD. (U.S.P. 1,687,228, 9.10.28. Appl., 21.12.22).—See B.P. 208,689; B., 1925, 114.

Dispersing agent [for dyes] and its manufacture. F. FELIX, Assr. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,680,778, 14.8.28. Appl., 23.5.27. Switz., 29.5.26).—See B.P. 271,898; B., 1928, 781.

Manufacture of aromatic amino-sulphocyanoderivatives. O. SPENGLER and W. MÜLLER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,687,596, 16.10.28. Appl., 28.4.26. Ger., 24.9.24).—See B.P. 240,420; B., 1925, 1012.

Preparation of alkyl ethers of 3'-nitro-4'-hydroxy-*o*-benzoylbenzoic acid. NEWPORT CO. (B.P. 273,342, 28.6.27. U.S., 28.6.26).—See U.S.P. 1,665,541; B., 1928, 474.

Manufacture of *N*-dihydro-1 : 2 : 1' : 2' - anthraquinoneazine. F. B. DEHN. From NEWPORT CO. (B.P. 297,692, 25.4.27).—See U.S.P. 1,634,473; B., 1927, 771.

Manufacture of carbamide (G.P. 448,200). Catalyst for methyl alcohol and formaldehyde synthesis (F.P. 606,596).—See VII. Sulphonic acids (B.P. 288,612).—See XII.

IV.—DYESTUFFS.

PATENTS.

Manufacture of water-soluble condensation products from amines or acid amides and acetaldehydedisulphonic acid. I. G. FARBENIND. A.-G., Assees. of T. MARIAM and W. BUTTENSCHÖN (G.P. 448,738, 8.4.25).—Bases are obtained in water-soluble form, from which the bases are readily regenerated by hydrolysis in presence of acid, by condensation with acetaldehydedisulphonic acid. Examples are methylamine, carbamide, *m*-aminophenol, dianisidine, chrysoidine. The chrysoidine compound dyes acetate silk reddish-yellow from a neutral bath. C. HOLLINS.

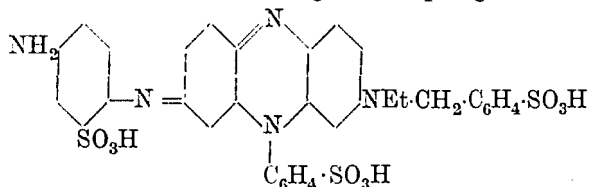
Manufacture of dyes containing chromium. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,616, 11.1.28).—Dyes formed by coupling a diazotised *o*-aminophenol, e.g., 4-nitro- or 4-chloro-*o*-aminophenol-6-sulphonic acid, with pyrazolones, e.g., 1-phenyl-3-methylpyrazolone, are heated under pressure with aqueous chromium formate at 120°. C. HOLLINS.

Manufacture of after-chromable dyes. I. G. FARBENIND. A.-G., Assees. of H. GROTHOWSKY (G.P. 447,754, 14.7.25).—An aromatic aldehyde is condensed with an equimolecular mixture of an *o*-hydroxycarboxylic acid and a heterocyclic compound containing a reactive CH₂ or CH group, and the product is oxidised. 2 : 6-Dichlorobenzaldehyde with *o*-cresotic acid and

dihydroxythiazole or phenylmethylpyrazolone gives a chrome-blue; benzaldehyde-*o*-sulphonic acid with *o*-cresotic acid and barbituric acid a chrome red-blue.

C. HOLLINS.

Manufacture of new direct dyes for cotton and for regenerated cellulose materials. BRIT. DYE-STUFFS CORP., LTD., and W. A. SILVESTER (B.P. 296,803, 7.6.27).—Acid dyes of the azine type, containing an external amino-group and preferably three sulphonic groups, are converted by phosgenation into direct dyes for cotton and viscose silks. Thus the dye of B.P. 13,406 of 1907 (annexed formula) gives on phosgenation a



bright blue dye; similarly, acid azine dyes made from 4-nitro-4'-aminodiphenylamino-2 : 3'-disulphonic acid, or 4-nitro-4'-amino-3-methyldiphenylamine-2'-sulphonic acid, may be phosgenated.

C. HOLLINS.

Manufacture of dyes of the anthracene series.

I. G. FARBENIND. A.-G. (B.P. 272,924, and Addn. B.P. 273,299 and 279,401, 15.6., 21.6., and 26.8.27. Ger., 15.6., 24.6., and 21.10.26).—(A) The sulphuric ester of leuco- β -aminoanthraquinone, prepared by reduction of 2-acetamidanthraquinone, treatment with chlorosulphonic acid and pyridine, and hydrolysis with alkali, is oxidised with alkaline permanganate to a violet acid wool dye, which is further oxidised with nitrous acid to indanthrone. (B) Oxidation of the leuco-ester with acid ferric chloride solution gives indanthrone directly. (C) The dimethyl ether of leuco- β -aminoanthraquinone is prepared by reduction of 2-acetamidanthraquinone with hydrogen and nickel, treatment with methyl sulphate and alkali, and hydrolysis with alcoholic hydrochloric acid of the 2-acetamido-meso-dimethoxyanthracene, m.p. 253°, so obtained. The ether is oxidised by acid ferric chloride solution to a compound, m.p. above 290°, from which by removal of methoxy-groups with concentrated sulphuric acid at 90–100° indanthrone is obtained.

C. HOLLINS.

Manufacture of condensation products of anthraquinone. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 296,817, 10.6.27).—Glyoxal, or a compound furnishing glyoxal, is condensed in acetic acid with anthrone or α -chloroanthrone to give glyoxylidenebisanthrones, which are yellow vat dyes.

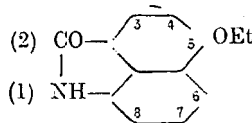
C. HOLLINS.

Manufacture of dye preparations and their application. SOC. CHEM. IND. IN BASLE (B.P. 293,795, 4.7.28. Switz., 12.7.27).—Bright blue-violet shades are obtained by using a mixture of isodibenzanthrone (brown-violet) and its dichloro-compound (red-violet). The dyes may be mixed in the form of powders, leuco-compounds, or leuco-ester salts.

C. HOLLINS.

Manufacture of new derivatives of anthranthrene. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 297,234, 25.10.27).—Hydroxy- and alkoxy-1 : 1'-dinaphthyl-8 : 8'-dicarboxylic acids, obtained from the corresponding naphthastyrils, are cyclised by means of

sulphuric acid or zinc chloride to give vat dyes. 5-Ethoxynaphthastyril (annexed formula) is boiled with alkali



and the resulting amino-acid is converted by diazotisation into 5 : 5'-diethoxy-1 : 1'-dinaphthyl-8 : 8'-dicarboxylic acid (cf. B.P. 276,126 and 278,100; B., 1927,

808, 902), which when treated with concentrated sulphuric acid yields 3 : 9-diethoxyanthanthrone, a reddish-orange vat dye; 3 : 9- and 2 : 8-dimethoxyanthanthrones (orange) are similarly obtained from 5- and 4-methoxynaphthastyrils, respectively. Naphthastyril-6-sulphonic acid is hydrolysed and diazotised to give 4 : 4'-disulpho-1 : 1'-dinaphthyl-8 : 8'-dicarboxylic acid, which is converted by alkaline fusion into the 4 : 4'-dihydroxy-compound; cyclisation of this with zinc chloride in acetic anhydride yields 4 : 10-dihydroxyanthanthrone; the dimethyl ether is a violet vat dye.

C. HOLLINS.

Manufacture of vat dyes. I. G. FARBENIND. A.-G., Assecs. of H. WOLFF (G.P. 447,556, 25.7.25).—Red-violet vat dyes are obtained by the action of chlorosulphonic acid on isodibenzanthrones in presence of selenium or its compounds.

C. HOLLINS.

Manufacture of new [yellow] vat dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,386, 30.5.27).—Yellow vat dyes are obtained by alkaline fusion of anthrapyrimidines or anthrapyrimidone [pyrimidanthrones or ketopyrimidanthrone]; the pyrimidone products are subsequently alkylated, e.g., with methyl *p*-toluenesulphonate. 2-Methylpyrimidanthrone is prepared from 2-amino-1-hydroxyanthraquinone by conversion first into the oxazole with acetic anhydride, and thence by the action of ammonia into 4-amino-2-methylpyrimidanthrone, and finally removing the amino-group.

C. HOLLINS.

Manufacture of [vat] dyes from dinaphthylene oxide. I. G. FARBENIND. A.-G., Assecs. of R. PUMMERER and E. PRELL (G.P. 449,121, 3.1.25).—Dinaphthylene oxide is oxidised in nitrobenzene and acetic acid with lead peroxide to a violet-brown vat dye, or in nitrobenzene with benzoyl peroxide to a grey (cotton) to black (wool) vat dye. Neutral oxidation with benzoyl peroxide gives a yellow compound which is converted by sulphuric acid into a brown-red vat dye. Oxidation with iron alum also gives a brown-red vat dye, whilst ozonisation in carbon tetrachloride leads to a red dye.

C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. I. G. FARBENIND. A.-G., Assecs. of O. BRAUNSDORF, P. NAWIASKY, and E. HOLZAPFEL (G.P. 448,262, 21.10.24).—Benzanthronyl thio-ethers are heated with alkali. 3 : 3'-Dibenzanthronyl sulphide or *p*-tolyl 3-benzanthronyl sulphide yields isodibenzanthrone; 2-methoxy-3 : 3'-dibenzanthronyl sulphide, m.p. 338–340°, gives methoxyisodibenzanthrone, a blue-violet vat dye; from 2 : 2'-dimethoxy-3 : 3'-dibenzanthronyl sulphide a blue vat dye results. Violet vat dyes are obtained by alkaline fusion of 8-amino-3 : 3'-dibenzanthronyl sulphide, the 8 : 8'-diamino-compound, mono- or dibrominated 3 : 3'-dibenzanthronyl sulphide.

C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. A. CARPMAEL. From I. G. FARBENIND. A.-G.

(B.P. 297,133, 11.6.27).—Aluminium chloride combines with pyridine to form a well-defined compound, soluble in excess of pyridine, which is an excellent condensing agent; less aluminium chloride is used, and reaction proceeds more smoothly at lower temperatures and without frothing. Other tertiary bases may be employed in place of pyridine. 4-Benzamido-1:1'-dianthraquinonylamine, heated at 125° with aluminium chloride (1.6 pts.) and pyridine (5 pts.), gives a brown vat dye. Similarly 1:2-dianthraquinonylamine gives an orange-yellow, 6- α -anthraquinonylamino-*N*-methylpyridoneanthrone a brownish-orange, 1:4-di-(α -anthraquinonylamino)anthraquinone a reddish-brown, and 2-aminoanthraquinone flavanthrone. (Cf. B.P. 278,771 and 288,291; B., 1928, 9.) C. HOLLINS.

Manufacture of indigoid vat dyes. I. G. FARBENIND. A.-G. (B.P. 282,805, 28.12.27. Ger., 24.12.26).—5-Chloro-7-methylisatin- α -5-chloro-*o*-tolylimine, prepared from 5-chloro-*o*-toluidine, is condensed with 5-chloro-7-methylthioindoxyl (bluish-violet), with 5:6:7-trichloro-thioindoxyl (violet), with 5:7-dichloro-4-methylthioindoxyl (violet-blue), with 6-chloro-4-methylthioindoxyl (violet), or with 5-chloro-4:7-dimethylthioindoxyl (bluish-violet). C. HOLLINS.

Manufacture of [indigoid] vat dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 297,376, 20.6.27).—Clear fast dyes, including navy-blue and red shades, are made by condensing a dihalogenomethylisatin, or its α -chloride etc., with naphthols, anthrols, indoxyls, thioindoxyls, etc. Examples are: 6-chloro-5-bromo-7-methylisatin α -chloride with 4-chloro- α -naphthol (navy-blue); 4-chloro-5-bromo-7-methylisatin α -chloride with α -anthrol (greenish-blue); 5:7-dichloro-6-methylisatin α -chloride with 6-chloro-4-methylthioindoxyl (reddish-violet), or reduced with hydrogen sulphide to 5:5':7:7'-tetrachloro-6:6'-dimethylindigo (reddish-blue); 5:7-dichloro-6-methylisatin with thioindoxyllic acid (red); 5:7-dichloro-4:6-dimethylisatin α -chloride with 4-chloro- α -naphthol (navy-blue). C. HOLLINS.

Manufacture of [thioindigoid] vat dyes. I. G. FARBENIND. A.-G. (B.P. 271,906, 30.5.27. Ger., 29.5.26).—5:5'-Dichloro-4:4':6:6'-tetramethylthioindigo, from 2-chloro-*m*-4-xyldine, m.p. 59–60°, 6:6'-dichloro-4:4':5:5'-tetramethylthioindigo, from *o*-3-xyldine by way of 5-chloro-2-cyano-3:4-dimethylphenylthioglycollic acid, and the condensation products of the corresponding thioindoxyls with isatins, thioisatins, acenaphthoquinone, etc. (especially with the *p*-dimethylaminoanils of 5:6-dichloro-4-methyl- and 6-chloro-4-methyl-thioisatins) are very fast to light and to kier-boiling. C. HOLLINS.

Manufacture of azo dyes. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 296,473, 1.6.27).—2:6-Dichloro-*p*-xyldine, m.p. 47–48°, or 5:6-dichloro-*p*-xyldine, m.p. 78–79°, obtained by chlorination of *p*-xyldine sulphate, are coupled in substance or on the fibre with 2:3-hydroxynaphthoic or acetoacetic arylamides. The former, e.g., 2:3-hydroxynaphthoic 5-chloro-*o*-toluidide or β -naphthylamide, give dyeings of good fastness to chlorine and kier-boiling. C. HOLLINS.

Production of metal compounds of azo dyes. I. G. FARBENIND. A.-G. (B.P. 272,908, 13.6.27. Ger., 15.6.26).—Water-insoluble *o*-hydroxyazo dyes are converted smoothly into their chromium or copper complex compounds if a large proportion of a solvent for the dye is added to the metal formate solution. Thus the dye from *p*-chloro-*o*-aminophenol and β -naphthol or phenylmethylpyrazolone is heated in an autoclave with about 4 pts. of aqueous chromium formate solution and about 10–11 pts. of ethyl alcohol. Other suitable solvents are acetone, glycol, ethylene chlorohydrin, polyglycol, etc. C. HOLLINS.

Manufacture of complex metal compounds of *o*-hydroxymonoazo dyes. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,819, 20.5.27).—Monoazo dyes from diazotised *o*-aminophenol ethers are boiled with chromium or copper formate under pressure at 125–130° so that the alkoxyl group is hydrolysed to hydroxyl. Examples are *o*-anisidine \rightarrow β -naphthol; 4-chloro-*o*-anisidine \rightarrow F-acid; 4-nitro-*o*-anisidine \rightarrow F-acid; *o*-anisidine-4-sulphonic acid \rightarrow β -naphthol; *o*-phenetidine \rightarrow Laurent acid. C. HOLLINS.

Production of azo dyes containing copper and chromium. I. G. FARBENIND. A.-G. (B.P. 272,949, 17.6.27. Ger., 17.6.26).—Chromium or copper compounds of azo dyes containing less than saturation amounts of the metal are treated with copper or chromium salts to produce complexes containing both metals; or the azo dye may be treated with a mixture of chromium and copper salts. Thus the dye, 4-nitro-2-aminophenol-6-sulphonic acid \rightarrow β -naphthol, gives with chromium salts alone a black, but with chromium and copper salts a bright violet. C. HOLLINS.

Stable diazo preparations. I. G. FARBENIND. A.-G., Assees. of K. SCHNITZSPAHN (G.P. 442,075 and 443,284, 19.6.25. Addns. to G.P. 426,033; B., 1926, 781).—Nitroarylamines, containing no sulphonic or carboxylic groups, in the form of their mineral acid salts (A) or free bases (B), are mixed dry with sodium nitrite and solid mineral acids or acid salts capable of liberating nitrous acid from nitrites. Partly dehydrated aluminium sulphate etc. or stabilising compounds such as sodium naphthalenedisulphonates may be added. Addition of sodium isopropyl naphthalenesulphonate improves the wetting properties. C. HOLLINS.

Stable diazo preparations. I. G. FARBENIND. A.-G., Assees. of K. SCHNITZSPAHN (G.P. 447,069, 28.12.24).—A nitroarylamine hydrochloride is mixed with sodium 1:6-naphthalenedisulphonate and sodium nitrite. C. HOLLINS.

Manufacture of dicyclic derivatives of pentamethylene. [1-Aryl-3:4-trimethylene-5-pyrazolone.] C. MANNICH (U.S.P. 1,685,407, 25.9.28. Appl., 15.10.26. Ger., 27.10.25).—See B.P. 260,577; B., 1927, 869.

Glyoxaldianthraquinone compounds. R. BERLINER, B. STEIN, and W. TRAUTNER, ASSRS. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,646,782, 25.10.27. Appl., 16.9.26. Ger., 30.10.25).—See B.P. 296,817, preceding.

Diazotisable dyes. M. BOENIGER, ASSR. to CHEM. WORKS FORMERLY SANDOZ (U.S.P. 1,686,947, 9.10.28.

Appl., 15.10.25. Ger., 28.3.25).—See B.P. 252,957; B., 1926, 657.

Benzanthrone condensation products. (B.P. 296,490).—See III. Lakes (B.P. 275,943).—See XIII. Photographic plates etc. (B.P. 292,140).—See XXI.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Behaviour in polarised light of threads from silk coagula of various stages of dehydration and age. P. P. VON WEIMARN (Kolloid-Z., 1928, 46, 36—38).—The author has studied the microstructure of a silk coagulum and its behaviour in polarised light during the alteration of its consistency and elastic properties with age, and the phenomena are compared with the reverse process, i.e., the swelling of natural silk threads. When natural silk threads swell in hot, concentrated sodium iodide solution the fibrils become better delineated and assume a spiral form, eventually lying perpendicular to the axis of the thread. Ultimately the fibrils liquefy to drops, which are at first spiral in form, and the mass becomes syrupy. In polarised light, the bright interference colours of the silk threads gradually disappear during swelling. To study the reverse process, a solution of silk in aqueous sodium iodide solution was coagulated by addition of concentrated sodium citrate solution to a syrupy consistency. On keeping, spiral forms can be observed under the microscope and later a thread-like structure appears; simultaneously, interference colours begin to be shown in polarised light, and these increase in brightness with age. Silk threads which have been prepared artificially in this way, and have been kept for 2 years, are not essentially different in properties from natural silk threads. E. S. HEDGES.

Physico-chemical nature of natural silk solutions in neutral, acid, and alkaline solvents. P. P. VON WEIMARN (Kolloid-Z., 1928, 46, 40—43).—The action of aqueous solutions of neutral salts on natural silk is compared with the effect of an excess of potassium iodide or silver nitrate on silver iodide. In the latter case a small excess of potassium iodide or silver nitrate peptises silver iodide to a sol, the particles of which dissolve in a large excess of the reagent to give a complex salt; the latter is decomposed on addition of water with formation of a sol once more. Precisely similar relations hold in the action of dilute and concentrated solutions of neutral salts on natural silk. The rôle of hydration in these phenomena is discussed, and a distinction is made between capacity and intensity of hydration. In solutions of acids or alkalis, the behaviour of silk is similar to that in neutral salt solutions only when the temperature is kept low; otherwise decomposition of the fibroin occurs and the regenerated substance differs from the original in composition. E. S. HEDGES.

Causes of the rubber-like state of matter. P. P. VON WEIMARN (Kolloid-Z., 1928, 46, 38—40).—Silk coagula with elastic properties can be obtained, and, examination of these has shown that they are characterised by a spiral formation of the fibrils, whilst the highly viscous liquid between the fibrils can act in the manner of a lubricating oil. Only under these conditions is elasticity observed in silk coagula, and when with age the spiral fibrils unite to form larger threads the

coagulum assumes plastic properties. By stretching, or through the syneresis which develops in time, the fibrils become oriented parallel to the axis of the threads and then the substance takes on solid properties. Threads of caoutchouc also possess the spiral form described above. When the caoutchouc has been subjected to stretching several hundreds of times in succession, the fibrils become oriented parallel to each other, but revert to the spiral form after resting.

E. S. HEDGES.

Microbiology of wool. R. BURGESS (J. Text. Inst., 1928, 19, 315—322 T).—The essential properties of an efficient antiseptic for the prevention of mildew in woollen goods are described. Whilst many substances such as sodium fluoride, chloramine-T, chlorinated phenols, etc. do not inhibit the growth of fungi and bacilli, others such as mercuric chloride, salicylic acid, thallium carbonate, etc., and notably sodium silicofluoride, give better protection. The protective effect of dyes is discussed, and chrome dyeing is found to afford considerable protection. The conditions of testing antiseptics, the effect of the alkalinity or acidity of the cloth under examination, and methods of detecting damaged fibres by staining tests are discussed.

B. P. RIDGE.

Determination of china clay in sized cotton goods. G. SMITH (J. Text. Inst., 1928, 19, 323—328 T).—A simple, direct method is described which depends on the fact that when sized cotton material is heated with hydrochloric acid (*d* 1.1), practically the whole of the inorganic material of the cotton is rendered soluble in water, whilst china clay is not appreciably attacked, and soluble salts of zinc, magnesium, calcium, and sodium present in the size are not affected. In order to allow for loss of combined water and of acid-soluble matter from the clay, a correction factor is applied to the weight of ignited clay found. The terms "natural," "dry," and "ignited" clay are defined, the possible sources of error in the method are discussed, and the conditions necessary for obtaining representative values are detailed. B. P. RIDGE.

New reagent for detection of oxycellulose. W. F. A. ERMEN (J. Soc. Dyers and Col., 1928, 44, 303—305).—Oxidised cellulose may be detected by staining blue with ferric ferrocyanide when immersed for 1 min. in a boiling solution of ferric ferrocyanide consisting of 250 c.c. of water and 5 c.c. each of solutions containing (per 100 c.c.) 20 g. of ferric sulphate and 25 g. of ammonium sulphate, and 33 g. of potassium ferrocyanide. Pure bleached cotton reacts slightly, and unbleached cotton more so with this reagent, but not sufficiently to interfere with the detection of oxidised cellulose. The reagent is readily reduced by sodium hyposulphite, phenol, aniline, tannic acid, wool, but not silk. Cotton may be dyed with Prussian Blue by first mordanting with tannic acid and tartar emetic, and then immersing in the cold reagent, whereas wool may be dyed directly. A suitable method for dyeing rich black shades free from brown on fur consists of mordanting the fur with Prussian Blue by treatment with the reagent and then dyeing it with *p*-phenylenediamine in the presence of an oxidising agent. Sulphur-black dyeings may be topped with Prussian Blue by

treatment with the reagent; similarly, sulphur-blues may be brightened and sulphur-yellows toned to bright greens. When treated together with the reagent, wood pulp becomes stained deep blue and cotton or linen paper remains almost unaffected. A. J. HALL.

X-Ray methods used in determining the structure of cellulose fibres. O. L. SPONSLER (Ind. Eng. Chem., 1928, 20, 1060—1062).—To avoid complications resulting from the presence of the central canal in cellulose fibres, small blocks of practically parallel fibres are prepared for section cutting. These may be rotated in the X-ray beam to secure longitudinal or transverse passage of the rays. A. G. POLLARD.

Decomposition of pine wood with sulphites. C. G. SCHWALBE and W. LANGE (Papier-Fabr., 1928, 26, 641—644).—The action of sodium sulphite solution on pine wood at 5—5½ atm. pressure continues for a period of 48 hrs. Preliminary soaking with sodium bisulphite decreased the necessary cooking period subsequently. Digestion of the wood shavings even at 170° after preliminary soaking still produced a harsh pulp. The preliminary soaking, whilst not incurring any serious chemical changes, is considered of importance in loosening incrusting material. Comparison of steam-digestion processes following preliminary impregnation with the bisulphites of sodium, calcium, and magnesium showed the last-named to be the most effective. In the production of wet-digested pulp, the best quality half-stuff was obtained by a short preliminary soaking in sodium bisulphite, followed by pressure-digestion with sodium sulphite for 8½ hrs. at 170°. In the steam-digestion trials the fibre was softened, but incrusting material was not satisfactorily removed; the pulp was characterised by high lignin content. A. G. POLLARD.

Influence of metallic salts on the tensile strength of artificial silk. J. EGGERT (Chem.-Ztg., 1928, 52, 794—795).—The general effects of the presence of metallic salts in bleaching and finishing baths for spun filaments are outlined and discussed. In particular, copper chloride increases the elasticity and decreases the tensile strength of both wet and dry filaments. This effect is not the result of over-bleaching produced by the activation of hypochlorite by the copper, but may be due to changes within the cellulose molecule. The importance of the prevention of contamination of bleaching baths by contact with metallic machine parts is emphasised. A. G. POLLARD.

Turbidity and gelatinisation of viscose caused by addition of chemicals. T. MUKOYAMA (J. Cellulose Inst., Tokyo, 1928, 4, 170—177).—A viscose solution (4.1% of cellulose and 3.4% of alkali) was treated with various salt solutions, and changes in turbidity and rate of gelatinisation were examined. The ammonium ion tends to accelerate turbidity formation and gelatinisation proportionally to the concentration used. Sodium chloride, sodium sulphate, and glycerin are much slower in their action, and their effect is less sensitive to changes in concentration. Sulphate and sulphite ions restrain the gelatinising effect of salts, especially when the sodium salts are used. A. G. POLLARD.

Paper pulp from logging waste in the Douglas fir region. A. H. HODGSON (Ind. Eng. Chem., 1928,

20, 819—821).—About 20% of the timber in this region is being left in the woods as waste; of this two fifths in the form of western hemlock, white fir, and Sitka spruce can be used for sulphite and mechanical pulp, whilst the remainder, Douglas fir, cedar, and pine, which has inferior pulping qualities, is best suited for lumber. F. R. ENNOS.

Importance of hydrogen-ion concentration in the sizing of paper. H. ROSCHIER (Pulp and Paper Mag., 1928, 26, 639—640, 679—682, 715—717, 745—748).—Experiments are described which show that under ordinary manufacturing conditions engine-sized paper contains both aluminium resinate and free resin (either of which is of itself an effective sizing agent) in relative amounts depending on the p_H of the stock. Thus, at p_H 3.7—3.9 the amount of aluminium resinate formed is comparatively small, whilst at p_H 4.5—5.0 the combined resin in the size is converted mainly into aluminium resinate. In the first case the mechanism of sizing is probably as follows: the fibre immediately fixes any aluminium resinate that is formed and in addition adsorbs aluminium ions, the positive charge resulting therefrom enabling it to attract and fix the negatively-charged free resin. Under these conditions a large proportion of the alum is carried away in the backwater. In the second case, i.e., at p_H 4.5—5, the aluminium resinate is fixed by the fibre, whilst the excess of alum is hydrolysed to aluminium hydroxide in association with a small quantity of sulphuric acid; aluminium hydroxide being positively charged adsorbs the negatively-charged free resin giving an adsorption complex of the same sign which in turn is adsorbed by the negatively-charged fibres. Under these conditions very little alum appears in the backwater. D. J. NORMAN.

Dispersion of rosin in paper sizing. P. DELCROIX (Pulp and Paper Mag., 1928, 26, 1049—1051).—In the preparation of rosin size by passing caustic soda solution through a tower containing lump rosin, the optimum concentration of the soda solution is 5.2 g./litre. At this concentration variations in the temperature and rate of flow of the solution through the tower do not materially influence the rosin content (40 g./litre) of the resulting size, provided that the temperature does not fall below 15°, and the rate of flow does not exceed a certain maximum value, approximately 15 litres per day per litre capacity of the apparatus. D. J. NORMAN.

Effect of beating on the sizing resistance of paper. J. BERGER (Papier-Fabr., 1928, 37, 561—565).—The sizing effect resulting solely from beating has been investigated by preparing a range of bleached sulphite-wood papers beaten for varying periods of time in a Lampén mill and comparing them as regards their sizing resistance (measured by various flotation tests) with resin-sized printing papers of varying degrees of hardness. The results indicate that sizing resistance increases slowly with the degree of beating (wetness) of the pulp up to a point and then rapidly increases with further beating, so that it is possible to prepare beaten sheets which, though containing no added sizing agent, show a better resistance to penetration by aqueous liquids than do fully resin-sized printing papers of the same

weight. In the flotation tests for sizing resistance the most satisfactory results were obtained with an ink bath or with a bath containing 0.1% caustic soda solution, using phenolphthalein in admixture with 9 times its weight of powdered sugar as a dry indicator. For soft-sized papers the caustic soda bath gave the more reliable results, but for hard-sized papers an ink bath was preferred owing to the action of the caustic soda on the sizing agent after prolonged contact.

D. J. NORMAN.

Measurement of sizing strength of paper.

B. SCHAPIRA (Papier-Fabr., 1928, 26, 609—617).—Methods of determining the sizing strength of paper are collected, described, and classified according to the factor on which the method depends, thus, (1) absorption of ink, (2) absorption of interacting liquids, such as, *e.g.*, ferric chloride and tannin solutions, (3) electrolytic conductivity, and (4) the action of water, *e.g.*, in capillary rise or in causing the curling up of floating specimens of the paper. Distinction is made between surface and interior sizing strength, and the methods of determining the latter are criticised. The best relative values are given by the dry indicator method of the Bureau of Standards, although, with few exceptions, good agreement is obtained between the results of this and of the curling method. The method of Stöckigt gives fairly uniform results, but its error increases in magnitude with increasing sizing strength on account of selective adsorption. The electrolytic and ink-floating methods are too unreliable.

B. P. RIDGE.

Determination of p_H value of paper. B. SCHÜLZE (Papier-Fabr., 1928, 26, 625—629).—The significance of hydrogen-ion concentration in determining acidity or alkalinity is discussed and general methods of determining p_H values are described. The p_H value of paper may be found either colorimetrically by the use of indicator papers, graded with respect to their p_H range, or of the Wulff colorimeter, or electrolytically by means of *E.M.F.* measurements using a quinhydrone electrode. For samples other than raw or parchment papers the colorimeter method is recommended, using an aqueous extract of the paper, whilst for the exceptions named either the electrolytic or the indicator paper method may be used on the paper itself, without extraction. Parchment papers have p_H 3—4.5, whilst other papers have p_H 4.5—9 according to the conditions of manufacture; reasonable agreement is shown between results obtained by the different methods used.

B. P. RIDGE.

Bisulphite liquors. STEWART.—See VII.

PATENTS.

Cotton fabric and its manufacture. E. GMINDER (U.S.P. 1,679,767, 7.8.28. Appl., 23.12.25. Ger., 17.9.24).—Cotton goods are carded to produce a slight nap, and are then treated with sodium hydroxide solution (*d* 1.11 or stronger) to curl the nap, which is finally roughened and oiled to produce a woolly appearance.

C. HOLLINS.

Manufacture of artificial silk. BRYSLKA, LTD., and F. W. SCHUBERT (B.P. 293,977 and 296,856, 15.7.27).—Difficulties attending the production of threads of uni-

form denier in the stretch-spinning of cuprammonium silk are obviated by placing in the spinning bath: (A) a volute taper funnel comprising a strip wound in the form of a volute spring so that the friction caused by the passage of the thread through the funnel causes fresh liquor to be drawn in through the space between the successive convolutions of the spring; (B) a spinning funnel wider at the bottom than at the top, slightly flared at the bottom, and, when two-liquor spinning is used, preferably provided at the top with an inverted, truncated conical member.

D. J. NORMAN.

Production of artificial silk and apparatus therefor. J. P. BEMBERG A.-G. (B.P. 275,637, 4.8.27. Ger., 5.8.26).—Strong uniform threads are obtained in the stretch-spinning of, *e.g.*, cuprammonium hydroxide solutions of cellulose if two or more groups of filaments are produced and caused to travel through the coagulating bath adjacent to but separated from each other until they have lost their tackiness, after which the groups of filaments are united into a single thread. As many funnels as groups of filaments may be provided in the spinning bath, in which case the funnels lead to a common outlet tube. Various types of apparatus are described.

D. J. NORMAN.

Production of artificial silk or films. S. OHSAKA (B.P. 297,047, 13.6.27).—A solution of cellulose in sodium hydroxide and carbon disulphide is mixed with not more than 20% of a protein or albumin solution associated with fatty oils. After ripening, the solution is spun into a coagulating bath, heated to 70—100°, and dried. The product has increased tenacity and elasticity both in the dry and wet state. [Stat. ref.]

F. R. ENNOS.

Manufacture of artificial threads by the bobbin process. O. SINDL (B.P. 297,618, 10.1.28. Ger., 15.11.27).—After leaving the coagulating bath the thread is wound on to a rotating bobbin, which moves vertically in and out of a washing bath. In its upward movement the bobbin, owing to its rotation, throws off the diluted coagulating liquid adhering to it, which is collected by a protective sheath and prevented from returning to the washing bath.

F. R. ENNOS.

Manufacture of artificial silk threads. ACETA GES.M.B.H. (B.P. 286,608, 5.3.28. Ger., 3.3.27).—In the dry-spinning process the threads are made to pass downwards in an ascending current of warm air in a cell subdivided by an adjustable constriction, both parts being independently connected at their upper ends to the suction pipe. The main portion of the drying air and solvent vapour is thus withdrawn at the upper end of the lower subdivision of the cell, so that only a comparatively small volume of air and vapour passes near the spinning nozzle.

F. R. ENNOS.

Artificial silk. E. HEYMANN (B.P. 297,364, 15.3.27).—To produce a thread of subdued lustre, a mixture of a non-volatile substance, *e.g.*, mineral, animal, or vegetable oils, aniline, etc., with a volatile substance, *e.g.*, benzene, is finely distributed throughout the spinning solution, and the resultant thread is treated for the removal of a part of the added substances by evaporation, in some cases *in vacuo*, or by means of solvents.

F. R. ENNOS.

Manufacture of viscose silk. I. G. FARBENIND. A.-G. (B.P. 290,231, 10.5.28. Ger., 10.5.27).—Viscose is spun into a coagulating bath containing not more than 0.1% of a zinc salt (zinc sulphate) whereby threads having finely-channelled surfaces are formed, which enhance the optical covering power and give the product a silvery, opaque appearance. F. R. ENNOS.

Manufacture of viscose. DU PONT RAYON Co., Assees. of G. W. BLANCO and C. HENNINGSSEN (B.P. 272,475, 23.5.27. U.S., 12.6.26).—Sheets of raw cellulose are steeped in caustic alkali solution while being held out of contact with each other as far as possible by suitable corrugations thereon, so as to permit free passage of the steeping liquor to all parts. The excess liquor is removed by pressure and re-used to steep other raw material, the presence of a relatively high percentage of hemicelluloses having no deleterious effect, but actually serving to accelerate the ripening operation. F. R. ENNOS.

Production of viscose. R. SAJITZ and E. POTT [CHEM. FABR. POTT & Co.] (B.P. 281,679, 30.11.27. Ger., 30.11.26).—Considerable latitude in the temperature and duration of ripening of the alkali-cellulose and of the composition of the precipitating bath is permissible if addition is made in one or more of the successive stages of the manufacture of viscose of small quantities of the following: salts of aromatic sulphonic acids; naphthalenesulphonic acids alkylated in the nucleus, their salts and other derivatives; sulphonates of hydroaromatic compounds, especially of the phenanthrene and anthracene series. F. R. ENNOS.

Preparation of viscose cellulose compounds. J. M. BERNARD (B.P. 273,280, 14.6.27).—Cellulose is mixed in a closed vessel with carbon disulphide, the excess of which is removed and recovered by draining and evacuation. After treatment of the product with sufficient sodium hydroxide solution to form cellulose xanthate, the residual carbon disulphide is removed by a further evacuation, and the product is dissolved in dilute sodium hydroxide solution. F. R. ENNOS.

Imparting a silk lustre to bodies produced from viscose and other aqueous cellulose solutions. WOLFF & Co., E. CZAPEK, and R. WEINGAND (B.P. 274,054, 21.6.27. Ger., 8.7.26).—Finely-powdered asbestos or asbestine is mixed with the cellulose solution, which is then worked up in the usual manner. F. R. ENNOS.

Manufacture of cellulose esters. I. G. FARBENIND. A.-G. (B.P. 291,360, 30.5.28. Ger., 30.5.27).—Cellulose is treated with a halide of a lower fatty acid, *e.g.*, acetyl chloride, in the presence of an organic base, *e.g.*, pyridine, and an indifferent organic medium, *e.g.*, chlorobenzene, which dissolves, at least partly, the reaction product of the acyl halide with the organic base formed during the esterification process, but which is not a solvent for the cellulose ester itself. F. R. ENNOS.

Manufacture of sulphonated cellulose derivatives. I. G. FARBENIND. A.-G. (B.P. 277,317, 29.8.27. Ger., 10.9.26).—Sodium-cellulose reacts with *o*- or *p*-sulphobenzyl chloride to give a water-soluble sulphobenzyl ether which may be isolated as sparingly soluble calcium or barium salt, or by performing the reaction in alcohol,

and evaporating the alcohol. The products are non-inflammable, and textiles may be fire-proofed by impregnation with the aqueous solution and precipitation of the calcium, barium, or aluminium salt within the fibre. C. HOLLINS.

Manufacture of thermoplastic materials. BRIT. CELANESE, LTD. (B.P. 282,723, 23.11.27. U.S., 29.12.26).—Cellulose acetate or other cellulose ester or ether, finely ground so that 80% passes 80-mesh, is mixed with one or more plasticisers and/or softening agents, *e.g.*, ethyl phthalate and/or triacetin, in the absence of volatile solvents. Colouring and filling materials may be subsequently added. F. R. ENNOS.

Method of washing cooked straw. DE W. C. AMERINE (U.S.P. 1,678,718, 31.7.28. Appl., 9.6.26).—Straw is cooked under pressure with lime and sulphur in a revolving spherical digester. When digestion is complete water is forced into the digester against the steam pressure through a tube running through a trunnion and extending one quarter round the inside of the digester. This tube is fixed to and rotates with the digester; the outlet pipe, running through the other trunnion, remains stationary. D. J. NORMAN.

Chemically treating and washing pulp. G. A. RICHTER and M. O. SCHUR, Assrs. to BROWN Co. (U.S.P. 1,683,262, 4.9.28. Appl., 19.4.27).—Cellulose pulp is digested in an alkaline liquor, bleached in hypochlorite, and superbleached in chlorine water. The pulp is washed after each treatment, fresh water being used for the superbleached pulp whilst the effluent wash-waters from the superbleached and bleached pulp are used for washing the bleached pulp and alkali-digested pulp respectively, the first contaminated portions of each washing being discarded if desired. F. R. ENNOS.

Producing a substance for obtaining a high gloss and pure white colour on paper and the like. SIEBENHIRTNER CHEM. IND.-WERK GES.M.B.H. (B.P. 287,559, 15.3.28. Austr., 24.3.27).—A pasty mixture of saponified stearin and heavy-metal salts, preferably zinc salts, is added to the paper pulp. F. R. ENNOS.

De-inking of paper. A. R. LUKENS, Assr. to RICHARDSON Co. (U.S.P. 1,680,949, 14.8.28. Appl., 22.6.25).—Old newspapers etc. are treated with warm 3% sodium carbonate solution for 30 min., and a sodium salt of a sulphonated hydrocarbon oil (0.5%) is added to facilitate the washing-out of the inky matter. C. HOLLINS.

Fulling wool and the like. J. NÜSSLEIN, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,685,680, 25.9.28. Appl., 13.8.26. Ger., 21.1.25).—See B.P. 270,333; B., 1927, 599.

Manufacture of cellulose compounds. L. LILIENFELD (U.S.P. 1,674,405, 19.6.28. Appl., 23.7.24. Austr., 4.4.24).—See B.P. 231,802; B., 1925, 913.

Manufacture of artificial materials [from cellulose derivatives]. L. LILIENFELD (U.S.P. 1,674,403, 19.6.28. Appl., 23.7.24. Austr., 4.4.24).—See B.P. 231,808; B., 1925, 914.

Manufacture of artificial materials [from *N*-substituted cellulose urethanes]. L. LILIENFELD (U.S.P. 1,674,404, 19.6.28. Appl., 22.5.26. Austr., 30.5.25).—See B.P. 248,994; B., 1926, 532.

Manufacture of cellulose thiourethane esters and ethers. L. LILIENTHAL (U.S.P. 1,674,402, 19.6.28. Appl., 17.9.25. Austr., 11.10.24.)—See B.P. 241,149; B., 1926, 47.

Apparatus for filtering solutions used in manufacture of artificial silk etc. W. P. DREAPER (U.S.P. 1,685,775, 2.10.28. Appl., 2.6.23. U.K., 22.6.22.)—See B.P. 207,214; B., 1924, 52.

Treatment of fabrics [containing cellulose esters or ethers]. G. H. ELLIS, Asst. to CELANESE CORP. OF AMERICA (U.S.P. 1,686,149, 2.10.28. Appl., 3.12.26. U.K., 27.7.26.)—See B.P. 280,989; B., 1928, 85.

[Vibration-damping] bearings, and applications thereof [for textile spindles]. BRIT. CELANESE, LTD., E. C. COPE, and E. KINSELLA (B.P. 297,875, 30.6.27).

Process and apparatus for dressing webs of fabric. H. MALY (B.P. 290,217, 19.4.28. Ger., 10.5.27).

Drying apparatus for laundry and dry-cleaning purposes. BRITISH-AMERICAN LAUNDRY MACHINERY CO., LTD. From AMER. LAUNDRY MACHINERY CO. (B.P. 297,515, 2.7.27).

Electric centrifuge for spinning artificial silk. BERGMANN ELEKTRICITÄTS-WERKE A.G., Assees. of F. BEIER (B.P. 285,011, 6.2.28. Ger., 8.2.27).

Method of treating cakes of artificial silk manufactured by the centrifuge-spinning process. (Sir) G. C. MARKS. From F. KÜTTNER A.-G. (B.P. 287,097, 14.3.28).

Oxygen-containing compounds (U.S.P. 1,681,156).—See VII. **Livering of cellulose lacquers** (B.P. 278,696).—See XIII. **Disinfectant** (G.P. 448,705).—See XXIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Valuation of protective agents [in wet processes for wool]. H. BLACKSHAW (J. Soc. Dyers and Col., 1928, 44, 297—300).—Three methods are described for the valuation of agents used for protecting wool from the deleterious action of alkaline solutions. In a quantitative method, wool is treated under conditions comparable with those used on a large scale, and the physical properties of the treated wool are determined by suitable mechanical apparatus such as a Goodbrand ballistic machine. In two qualitative methods wool may be subjected to an exaggerated treatment such that the effect of a protective agent is more definitely shown by mechanical tests, or loose wool is treated under normal large-scale conditions, and the damage to the wool determined by the resulting increase in its affinity for Acid Scarlet 4R extra. Typical results obtained with Protectol are given.

A. J. HALL.

Influence of heat on the affinity of cotton for dyes. C. K. PATEL (J. Soc. Dyers and Col., 1928, 44, 301—303).—The affinity of cotton for Chlorazol Sky Blue FF, Benzopurpurine 4B, and Methylene Blue was determined after heating in an air oven at 150—285° for various periods of time, it being observed that the heated cotton has a smaller affinity for both direct and basic dyes. The loss of affinity for direct dyes is proportional to the duration and temperature of heating,

but for basic dyes the initial loss of affinity is large compared with further losses. It is suggested that part of the lustre of pile fabrics which have been singed is due to the comparatively small absorption of dye by the tips of the fibres which have been most affected by the heat of singeing.

A. J. HALL.

Method for increasing the affinity of cotton and other fibres for colouring matters. G. E. HOLDEN (J. Soc. Dyers and Col., 1928, 44, 305—307).—Cotton acquires an increased affinity for direct, sulphur, vat, water-solubilised vat, and basic (after mordanting with tannic acid or Katanol O) dyes when impregnated with a 5—20% solution of gelatin, then steamed for about 1 hr. under 5 lb. pressure, and afterwards freed by washing from the 82—83% of gelatin not thereby rendered insoluble (cf. Holden, B., 1927, 565). The increase of affinity is greater than that produced by mercerisation, and is directly proportional to the amount of gelatin fixed on the cotton; the resulting dyeings are better penetrated, faster to washing, and equal in fastness to light to those produced on cotton not prepared with gelatin. Multi-tone effects may be obtained by dyeing cotton fabric printed with solutions of gelatin of different concentrations. The insoluble gelatin in cotton prepared with gelatin is fast to soap, alkalis, acids, and sodium hyposulphite, but is removed by treatment with solutions containing free chlorine; fabric prepared with gelatin may be discharged by printing with a thickened 10% solution of chloramine-T and then steaming. Glue is not a satisfactory substitute for gelatin, but isinglass is superior. Harshness in handle of cotton materials prepared with gelatin may be removed by soaping at 80°.

A. J. HALL.

Effect of light on coloured [cotton] fabric. II. (Miss) E. HIBBERT (J. Soc. Dyers and Col., 1928, 44, 300—301; cf. B., 1927, 840).—Cotton fabric, particularly that having a pile surface, dyed with *o*-tolueneazo- β -naphthol became covered with bright lustrous crystals after exposure for 3 min. to light from a fadeometer (the temperature of the air surrounding the fabric was about 45°); the crystals were shown to be capable of further sublimation, and when recrystallised from chloroform were obtained as crystals having a different character but the same m.p. (133°) as that of *o*-tolueneazo- β -naphthol. A thin layer of powdered *o*-tolueneazo- β -naphthol sublimed only to a very small degree when heated to 45°, and it was not possible to obtain crystals as in the case of dyed fabric. When faded by exposure in a fadeometer, cotton fabric dyed with *o*-tolueneazo- β -naphthol suffers a loss of depth of shade due to sublimation, but its colour does not change or become dull as when exposed to sunlight. Sublimation effects were observed with cotton dyed with *o*-tolueneazo- α -naphthol and with combinations of Naphthol AS-BO and AS-RL with Fast Orange G base, but no crystals were formed on the surface of the fabric.

A. J. HALL.

Reagent for oxycellulose. ERMEN.—See V.

PATENTS.

Printing with developing dyes, especially on acetate silk. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,374, 20.6.27).—Acetate silk is printed with a solution of the dye components in thiodiglycol, tri-

(β -hydroxyethyl)amine, cyclohexyldi-(β -hydroxyethyl)-amine (B.P. 297,484; B., 1928, 846), or other suitable solvent, with thickening, and developed in an acidified nitrite bath with or without previous steaming; or nitrite may be added to the printing paste and the colour developed by acid alone. Examples of printing pastes are: β -naphthylamine, resorcinol, and nitrite in thiodiglycol (orange); 2-chloro-*p*-anisidine, 2:3-hydroxynaphthoic acid, and nitrite in cyclohexyldi-(β -hydroxyethyl)amine (bluish-red); dianisidine and 2:3-hydroxynaphthoic acid in cyclohexyldi-(β -hydroxyethyl)amine (blue-black). C. HOLLINS.

Oxidation process in dyeing or printing of textiles [with aniline black]. MANCHESTER OXIDE CO., LTD., R. H. CLAYTON, and F. SCHOLEFIELD (B.P. 296,530, 4.8.27).—In dyeing and printing with aniline black the ferrocyanide oxygen carrier is replaced by sodium carbonylferrocyanide. Greater stability at 15° is claimed, with increased activity at 100°; premature oxidation is thus minimised. C. HOLLINS.

Dyeing and printing of vegetable and animal fibres with basic dyes. E. I. DU PONT DE NEMOURS & Co. (F.P. 620,746, 6.8.26).—Dyes which are fixed by the fibre without steaming or mordants are obtained by condensing basic dyes (e.g., methylene-blue) in the form of free colour base with aromatic hydroxy-acids (tannin, β -resorcylic acid, salicylic acid) in presence of mineral acids, such as phosphoric, hydrochloric, sulphuric, and boric acids. The condensation products are salted out from the neutralised reaction mixture. C. HOLLINS.

Treatment of filaments, threads, yarns, and fabrics containing them of artificial silk having for basis cellulose esters or ethers [to produce discharge effects]. G. B. ELLIS. From Soc. FABR. DE LA SOIE "RHODIASETA" (B.P. 297,186, 26.7. and 31.10.27).—Dyes (except developed and ice colours) on acetate silk are discharged by application of adsorbent substances, such as animal charcoal, kaolin, fuller's earth, or infusorial earth, either in aqueous suspension or particularly in printing pastes. The action is enhanced by addition of swelling agents and by heat (e.g., by steaming after printing). C. HOLLINS.

Production of dyes or coloured substrates. I. G. FARBERIND. A.-G., Assees. of E. MÜNCH (G.P. 448,909, 24.4.25).—Indigoid dyes are regenerated by hydrolysis of the bisulphite compounds of their *N*-acetyl derivatives in presence or absence of a substrate. Cotton, e.g., is padded with a solution of *NN'*-diacetylindigo bisulphite compound and developed in an alkaline bath containing a little hydrogen peroxide. Wool is padded with the 4:4'-dichloro-compound in presence of acetic acid and sodium sulphate, and developed with dilute sodium hydroxide or ammonia with addition of hydrogen peroxide and sodium sulphate. Wool, padded with the bisulphite compound of *N*-acetyl-3-indole-2-thionaphthenindigo, is developed with dilute sulphuric acid and nitrite. By addition of sodium aluminate solution and sulphite-cellulose liquor to the bisulphite compound of *NN'*-diacetylindigo, colloidal indigo is precipitated, and a blue lake is obtained on acidification with acetic acid. The process may also be applied to printing. C. HOLLINS.

Dyeing [regenerated cellulose material]. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, R. BRIGHTMAN, and P. CHORLEY (B.P. 296,845, 7.6.27).—2:2'-Disubstituted benzidines (other than 2:2'-dinitro-) are tetrazotised and coupled with 2 mols. of a coupling component or of two different coupling components. Examples are: 2:2'-dichlorobenzidine with salicylic acid and γ -acid (orange-brown) or with 2 mols. of γ -acid (maroon); *m*-tolidine with salicylic acid and γ -acid (maroon) or phenyl- γ -acid (brown); benzidine-2:2'-dicarboxylic acid with 1-*p*-sulphophenyl-3-methylpyrazolone and β -naphthol (orange); benzidine-2:2'-disulphonic acid with 2 mols. of 1-phenyl-3-methylpyrazolone (yellow). Not more than two sulphonic groups should be present in the finished dye. C. HOLLINS.

Reserves on silk. I. G. FARBERIND. A.-G., Assees. of W. SCHLEGEL (G.P. 448,847, 6.12.23. Addn. to G.P. 432,111; B., 1927, 214).—The sulphurised phenols (syntans) applied in the prior patent for reserves on wool are now used, with or without addition of tin salts, for reserving silk, which remains white (or coloured) when admixed cotton or artificial silk is dyed with direct or basic colours. C. HOLLINS.

Reserve dyeing on vegetable fibres. H. LEEMANN and G. TAGLIANI, Assrs. to MUNITEX CORP. (U.S.P. 1,679,477—1,679,481, 7.8.28. Appl., 20.5.26. Ger., 7.5.24).—Cotton is locally reserved against direct dyes by printing with alkali and treating with an acid chloride (toluene-*p*-sulphonyl chloride). The reserved parts may be dyed (A) with a galloeyanine, (B) with acid dyes, (C) with acid dyes in the same bath as direct dyes for the unreserved parts, (D) with an arylamine which is subsequently diazotised and coupled with a developer, (E) in batik effects by means of a wax covering and basic and direct dyes. C. HOLLINS.

Production of yellow, orange, and brown coloured patterns on cotton or silk materials with the aid of wax reserves. I. G. FARBERIND. A.-G., Assees. of H. EICHWEDE and C. ZECHENTMAYER (G.P. 446,542, 19.12.24).—The wax-patterned material is padded with phosgenated 1-*p*-aminophenyl-3-methylpyrazolone, drained, and coupled with diazotised nitroaniline for yellow. Padding with the dye, benzidine-3:3'-disulphonic acid = 2 mols. of 1-*p*-aminophenyl-3-methylpyrazolone, and developing with diazotised *o*-nitroaniline gives an orange, which may be diazotised on the fibre and coupled with phenylmethylpyrazolone (orange). For orange-brown the material is padded with the dye, salicylic acid \leftarrow benzidine-3-sulphonic acid \rightarrow 1-*p*-aminophenyl-3-methylpyrazolone, and developed with diazotised *o*-nitroaniline. All the couplings proceed smoothly in the cold. C. HOLLINS.

Modifying the capacity of cellulose-containing materials for taking up colours. HERMINGHAUS & Co., G.M.B.H., and K. L. WENZ (B.P. 281,696, 1.12.27).—The successive treatment of cellulose fibres with a concentrated caustic alkali and an inorganic oxychloride in an inert solvent for conferring on such fibres an increased affinity for basic dyes and a decreased affinity for direct dyes (cf. B.P. 192,173; B., 1923, 304 A) is modified in so far as the concentrated alkali is replaced by alkali solutions not exceeding 3%; the

fibres do not suffer appreciable shrinkage under these conditions. A. J. HALL.

Production of azo dyes on the fibre. I. G. FARBENIND. A.-G. (B.P. 292,100, 5.6.28. Ger., 13.6.27).—The diazo component is applied in the form of a diazonium fluoborate. Enhanced brilliance is claimed.

C. HOLLINS.

Dyeing. P. MIJER, ASSR. to TWO-TONE CORP. (U.S.P. 1,683,687, 11.9.28. Appl., 14.2.24).—The liquid colour is atomised and deposited as a cloud in minute non-spotting particles on material placed below.

F. G. CLARKE.

Method of ornamenting textile pile fabrics. H. LIVSEY, G. E. HOLDEN, and J. & J. M. WORRALL, LTD. (B.P. 297,607, 17.12.27).—Pile fabrics are printed with solutions of cellulose, such as viscose and cuprammonium solutions, then dried, steamed, and washed to regenerate the cellulose.

A. J. HALL.

Treating and wetting-out fibrous material. Carbonising woollen fibres. R. H. POTT, ASSR. to CHEM. FABR. POTT & Co. (U.S.P. 1,686,836—7, 9.10.28. Appl., [A] 23.9.25, [B] 26.7.24. Ger., [A, B] 10.7.23).—See B.P. 248,814; B., 1926, 403.

Production of fast dyeings on the fibre. T. KIRCHEISEN and J. GÜRTLER, ASSRS. to GRASELLI DYE-STUFF CORP. (U.S.P. 1,680,791, 14.8.28. Appl., 21.1.27. Ger., 23.1.26).—See B.P. 262,537; B., 1927, 104.

[Apparatus for] dyeing textile goods. I. G. FARBENIND. A.-G. (B.P. 270,307, 27.4.27. Ger., 27.4.26).

Apparatus for the dyeing or treating of hanks. K. S. MILLS (B.P. 297,755, 22.3.27).

Dye preparations (B.P. 293,795).—See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Evaporating system for salt. A. MITTENBERG (Ukraine Chem. J., 1928, 4, [Tech.], 43—64).—A detailed analysis of the thermal economy of brine evaporating plants is given.

R. TRUSZKOWSKI.

Determination of iron carbonyl. R. H. GRIFFITH and G. C. HOLLIDAY (J.S.C.I., 1928, 47, 311—312 τ).—Gases containing iron pentacarbonyl are passed through strong sulphuric acid, which is then evaporated to dryness, the residue redissolved, and the iron determined as Prussian-blue by a colour-density method. If the gas contains unsaturated hydrocarbons, sulphuric acid cannot be used, and a method of determination by absorption in active charcoal is described. Examples of the carbonyl content of various gases are given.

Preparation of calcium and magnesium bisulphite liquors. D. W. STEWART (Pulp and Paper Mag., 1928, 26, 1013—1016).—The difficulties associated with the preparation of bisulphite liquor containing both lime and magnesia by the ordinary tower system can be obviated by adding pulverised calcined magnesite (temperature of calcination about 900°) to the water-feed at the top of an absorption tower containing limestone. By regulating the addition of the calcine, liquors are readily obtained with a lime-magnesia ratio as high as 0.64 : 1, owing to the solubility of the pulverised calcine.

D. J. NORMAN.

Manufacture of [solid] carbon dioxide. H. E. HOWE (Ind. Eng. Chem., 1928, 20, 1091—1094).—The production of gases of combustion containing 16—18% CO₂ from coke involves heavy renewal of boiler settings. The gases are scrubbed with water in steel towers packed with limestone and absorbed by 2.2*N*-sodium carbonate in high coke towers. The exit gases still contain 8% CO₂. The towers operate at 49° and the lye boilers at 115°. These boilers work at 7—8 lb. pressure, as fixed by the efficiency of the heat exchangers. The gas evolved is cooled and, after separation of oil and water, is compressed in three stages to 1100 lb./in.² The compressed gas is passed through permanganate and cooled to 21°, when it liquefies. The liquid is expanded to a pressure of 15 lb., from 50 to 60% evaporates and is returned, and the remainder solidifies. It is moulded, pressed, and wrapped in heavy paper.

C. IRWIN.

Determination of small quantities of carbonate in presence of excess of sulphide and chloride, with particular reference to the analysis of metallic corrosion products. W. H. J. VERNON and L. WHITBY (J.S.C.I., 1928, 47, 255—259 τ).—The ordinary gravimetric method for determination of carbonate is critically examined. Errors due to presence of sulphide or chloride are eliminated by interposing powdered copper or *p*-nitrosodimethylaniline respectively in the absorption train. By means of these reagents a large excess of hydrogen sulphide or hydrogen chloride may be quantitatively separated from very small quantities of carbon dioxide; it is shown that the reagents commonly recommended for the purpose are quite inadequate. The copper powder should precede the *p*-nitrosodimethylaniline; to ensure freedom from action on carbon dioxide it should not have been prepared by reduction with hydrogen. Phosphoric acid is used for decomposing the material in the evolution flask. "Sofnolite" is used for absorption of carbon dioxide. The accuracy obtainable is such that the observed weight-increment due to carbon dioxide is within 0.1 mg. of the calculated value, even when sulphide and chloride are simultaneously present in large excess. Small quantities of sulphide are determined by evolution as hydrogen sulphide, absorption in cadmium acetate solution, and titration with iodine and thiosulphate; to obtain a quantitative yield of hydrogen sulphide in presence of excess of copper salts, stannous chloride is added to the mixture in the evolution flask. The hygroscopicity of corrosion products is referred to; a curve is given showing the rate of removal of hygroscopic moisture from a sample of corrosion product (60 : 40 brass) on heating at 103°.

Detection of traces of carbonate. J. RAE (Pharm. J., 1928, 121, 315).—The substance is treated with dilute sulphuric acid and 2 c.c. of chloroform in a small flask, fitted with a rubber stopper carrying a thistle funnel with a two-bulb trap in which are placed 2—3 c.c. of lime water. Heating on the water bath causes the chloroform to distil, bringing with it the carbon dioxide; a turbidity in the lime water is apparent if the sample contains 1 mg. of sodium carbonate or its equivalent.

A. R. POWELL.

Fermentation carbon dioxide. WANDERSCHECK.
—See XVIII.

PATENTS.

Production of hydrocyanic acid. M. J. BROWN and E. A. HARDING, ASSRS. to ROESSLER & HASSLACHER CHEM. CO. (U.S.P. 1,680,662, 14.8.28. Appl., 19.10.25).—Sodium cyanide, or crude material containing it, is stirred with a solution of sodium hydrogen carbonate and vacuum is applied so that the water is vaporised at 35–45°. Hydrogen cyanide is carried away with the steam and is separated by known means.

C. HOLLINS.

Manufacture of oxygen-containing compounds of the aliphatic series [oxalic acid]. I. S. MELLANOV, ASSR. to KEMIKAL, INC. (U.S.P. 1,681,156, 14.8.28. Appl., 13.3.28).—Waste paper etc. is pulped and heated to 200° with caustic alkali, air being injected into the mass. Paraffin is added, and the temperature, after being raised to 210°, is finally maintained for some hours at 150° without air-injection. Sodium oxalate is produced.

C. HOLLINS.

Manufacture of pure tartaric acid and tartrates. SOC. ENO-TARTARICA ITALIANA (F.P. 624,540, 13.11.26. Italy, 17.11.25).—To tartar and aluminium hydroxide hydrochloric acid is added in such amount that the alumina is retained in solution on boiling. Pure potassium hydrogen tartrate crystallises out when cold. Calcium hypochlorite and aluminium sulphate are added to decolorise the solution. The potassium hydrogen tartrate is heated with water and sulphuric acid and potassium removed as potash alum by addition of alum. The centrifuged solution is evaporated *in vacuo* to $d\ 1.374$ and tartaric acid crystallises out.

C. HOLLINS.

Manufacture of alkali and ammonium phosphate. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,546, 27.7.27).—Dicalcium phosphate is treated with alkali fluoride or ammonium fluoride solution, the resulting calcium fluoride is removed by filtration, and the solution is concentrated or cooled after the addition, if desired, of phosphoric acid or fixed or volatile alkali. The mother-liquor is returned to the process. Alternatively, precipitation of the ammonium phosphate from the above filtrate may be effected by means of methyl alcohol.

W. G. CAREY.

Cyanides and [their manufacture from] cyanamide salts. FABR. DE PROD. CHIM. DE THANN ET DE MULHOUSE (Addn. F.P. 31,723, 26.11.25. to F.P. 586,110. Cf. B.P. 225,160; B., 1925, 98).—In the prior process the alkaline-earth carbide may be replaced by carbides of other metals (e.g., aluminium) or by alkali- or alkaline-earth metals, or magnesium, aluminium, or zinc. Certain oxides such as litharge or antimony trioxide, or certain alkali salts, such as sodium chloride, may be used in place of alkali carbonates.

C. HOLLINS.

Drying of gases containing ammonia and carbon dioxide, especially of gas mixtures circulating in the manufacture of carbamide. I. G. FARBENIND. A.-G.; Assees. of R. GRIESSBACH and M. SCHMIDT (G.P. 448,200, 3.2.25).—Alumina gel, prepared according to G.P. 436,671 (B., 1927, 482) and dried at red heat, absorbs water from mixtures of ammonia and carbon

dioxide. The mixture of carbon dioxide, ammonia, and water, obtained by heating ammonium hydrogen carbonate, is passed, for example, through a tower filled with alumina gel, and yields 80–90% of the ammonia as carbonate. The alumina may be replaced by a strongly heated mixture of bauxite and active silicic acid or base-exchanging substances. The process is of special importance for the drying of the wet gases from the working-up of the carbamide melt before returning them to the cycle. It is also applicable in the manufacture of methylamine from carbon dioxide, ammonia, and hydrogen. The drying agent may be regenerated after use by heating in a current of dry air or dry waste gases.

C. HOLLINS.

Preparation of a copper catalyst for synthesis of methyl alcohol and formaldehyde from carbon monoxide and hydrogen. SOC. NAT. DE RECHERCHES SUR LE TRAITEMENT DES COMBUSTIBLES (F.P. 606,596, 27.2.25).—A boiling solution of copper sulphate or nitrate, free from impurities (especially iron), is treated with caustic alkali, and the black hydrated oxide, washed free from alkali and dried in a vacuum at 50°, is mixed with 10–20% of the violet modification of metallic copper (Sabatier) and reduced at the ordinary pressure by hydrogen or carbon monoxide in a slow stream or diluted with nitrogen or methane not above 130–140°. The resulting porous, fine-grained, pyrophoric catalyst converts hydrogen and carbon monoxide at 275–300° and 100–200 atm. into methyl alcohol and formaldehyde.

C. HOLLINS.

Production of aluminium compounds from raw materials. CLAY REDUCTION CO. (B.P. 279,515, 25.10.27. Norw., 25.10.26).—Material containing oxygen compounds of aluminium and silicon, e.g., clay, is treated with a solution of ammonium fluoride above 34°, the water is evaporated, and the mixture heated at about 300° to remove volatile compounds of ammonium and silicon with fluorine, which are treated with ammonia and water to form silica gel and ammonium fluoride. The residue of complex metallic fluorides, after reduction of the iron with producer gas, is heated at 400–500° with dry hydrochloric acid and ammonia to convert the fluorides into chlorides, with formation of aluminium chloride and ammonium fluoride which are volatilised and separated by fractional condensation.

W. G. CAREY.

Refining of crude arsenic trioxide. C. L. READ, ASSR. to AMER. SMELTING & REFINING CO. (U.S.P. 1,681,496, 21.8.28. Appl., 14.4.26).—The volatilised arsenic trioxide is passed through baffled flues, maintained at such a temperature that arsenic trioxide is in part deposited in the vitreous condition. The latter absorbs the antimony present in the fume as impurity, and the pure arsenic trioxide passes on and is condensed. The volume of air passing through the furnace and flue is regulated in order to control the temperature and time of contact of the fume with the vitreous oxide.

F. G. CLARKE.

Recovery of hafnium. SIEMENS & HALSKE A.-G., Assees. of B. FETKENHEUER (G.P. 446,489, 26.3.26).—The hafnium-rich mother-liquors obtained in the fractionation of double fluorides of zirconium and hafnium are treated with methyl or ethyl alcohol to

precipitate the double fluorides present, and the residual solution is neutralised with alkali or treated with further quantities of alkali fluoride to precipitate the remainder of the hafnium.

A. R. POWELL.

Manufacture of hydrogen. I. G. FARBERIND. A.-G. (B.P. 267,535, 10.3.27. Ger., 10.3.26).—Hydrocarbons, alone or mixed with other gases which do not contain free oxygen, are treated with steam at 300–600° in the presence of nickel, cobalt, or iron activated with chromium, vanadium, potassium, magnesium, aluminium, or alkaline-earth metals, the resulting carbon dioxide then being removed. The reaction may be performed in stages by transforming some of the hydrocarbons, removing the carbon dioxide, and repeating the succession of operations with the hydrocarbon–hydrogen mixture until all the hydrocarbons are converted.

W. G. CAREY.

Production of hydrogen. M. CASALE-SACCHI (B.P. 297,135, 13.6.27).—A mixture of oxygen and carbon dioxide, with or without nitrogen, is passed over a carbonaceous fuel heated at above 1000°, readily soluble or absorbable catalyst poisons are removed, and the resulting carbon monoxide is treated with steam in the presence of a catalyst, *e.g.*, a metal of the iron group, and the carbon dioxide is separated from the hydrogen, part of the carbon dioxide being used for further admixture with oxygen.

W. G. CAREY.

Treatment of sulphur. J. W. SCHWAB, Assr. to TEXAS GULF SULPHUR CO. (U.S.P. 1,683,731, 11.9.28. Appl., 27.12.26).—Abnormally coloured sulphur has its normal colour restored by treatment of the molten material with finely-divided activated carbon, from which it is afterwards separated.

H. ROYAL-DAWSON.

Purification of gases [in synthesis of ammonia]. G. F. UHDE (U.S.P. 1,685,733, 25.9.28. Appl., 15.4.26. Ger., 9.2.25).—See B.P. 247,226; B., 1926, 979.

Synthetic production of ammonia. G. F. UHDE (U.S.P. 1,685,734, 25.9.28. Appl., 28.5.26. Ger., 4.6.25).—See B.P. 253,122; B., 1927, 481.

Apparatus for production of synthetic ammonia. G. FAUSER (U.S.P. 1,686,371, 2.10.28. Appl., 21.8.25. Italy, 24.9.24).—See B.P. 240,436; B., 1926, 979.

Apparatus for synthesis of ammonia. G. CLAUDE, Assr. to LAZOTE, INC. (U.S.P. 1,686,799, 9.10.28. Appl., 22.12.21. Renewed 17.12.26).—See B.P. 174,041; B., 1923, 144 A.

Carrying-out [exothermic] gaseous catalytic reactions [for synthesis of ammonia]. R. E. SLADE, Assr. to ATMOSPHERIC NITROGEN CORP. (U.S.P. 1,686,349, 2.10.28. Appl., 1.5.26. U.K., 4.5.25).—See B.P. 255,964; B., 1926, 822.

Precipitating heavy metals from ammoniacal solutions. C. MÜLLER, L. SCHLECHT, and W. SCHUBARDT, Assrs. to I. G. FARBERIND. A.-G. (U.S.P. 1,686,391, 2.10.28. Appl., 11.3.27. Ger., 9.4.26).—See B.P. 269,164; B., 1927, 907.

Methods of obtaining zirconium compounds. TITANIUM ALLOY MANUF. CO., Assees. of C. J. KINZIE

(B.P. 271,873, 23.5.27. U.S., 27.5.26).—See U.S.P. 1,658,807; B., 1928, 297.

Apparatus for utilising impure gases or exhaust gases containing carbon dioxide. F. RIEDEL, Assr. to RIEDEL FERTILISING PROCESS CO. (U.S.P. 1,687,229, 9.10.28. Appl., 8.3.22. Ger., 14.3.16).—See G.P. 315,019; B., 1920, 489 A.

Sodium acetate (F.P. 583,341).—See III. **Anti-corrosive silver alloys** (B.P. 297,165).—See X. **White lead** (B.P. 273,287).—See XIII. **Solidified iodine solutions** (B.P. 277,953).—See XX.

VIII.—GLASS; CERAMICS.

Heat balance of a glass tank furnace. R. D. PIKE and G. H. WEST (J. Amer. Ceram. Soc., 1928, 11, 734–744).—A method is described for determining the thermal balance of an oil-fired, continuous, regenerative, glass tank furnace. Means for reducing the fuel consumption by 14% are indicated. Considerable loss of heat through the furnace brickwork was discovered.

F. SALT.

Waste-heat drying system involving recuperation. W. T. WINDSOR and F. C. WESTENDICK (J. Amer. Ceram. Soc., 1928, 11, 730–733).—In order to eliminate smoke and utilise waste heat from a brick kiln for drying, a “double-deck” system of ducts was arranged leading to the kiln outlet. By means of dampers the upper duct was made to take the waste heat from cooling kilns, and the lower duct to take the combustion gases. The waste heat was discharged by a fan into a main duct connecting with each tunnel.

F. SALT.

Dorr classifiers for clay-washing. A. ANABLE (J. Amer. Ceram. Soc., 1928, 11, 791–794).—The Dorr bowl classifier is described, and its advantages over the old method of using settling channels in washing clays, particularly primary and secondary kaolins, are pointed out.

F. SALT.

Vacuum treatment of clay slips and bodies. H. M. KRANER and A. H. FESSLER (J. Amer. Ceram. Soc., 1928, 11, 725–729).—Simple apparatus is described for determining the amount of gas removable from ceramic slips by subjecting them to reduced pressure. In a factory porcelain slip from 1 to 1.3% of air by vol. was found. The removal of this adsorbed air from the slip had practically no effect on the dielectric and mechanical strength of the fired porcelain.

F. SALT.

Firing terra cotta. S. J. McDOWELL and R. M. MURPHY (J. Amer. Ceram. Soc., 1928, 11, 745–752).—Tests were made on a 14 ft. periodic, coal-fired muffle kiln having no centre stack. Temperature and draught readings were taken, and gas analyses were made. A marked difference in the top and bottom temperatures in the muffle was observed; this was reduced from 300° to 100° maximum by the construction of a stack, 18 in. in diam., within the muffle. Kiln and operating data are given.

F. SALT.

Pores in bricks. G. J. EASTER (J. Amer. Ceram. Soc., 1928, 11, 764–768).—A number of standard refractory materials were tested for air permeability in a “permeameter” (*cf.* Westman and Pfeiffer, B., 1926, 917). The pores are arbitrarily considered as replaced

by parallel capillaries extending through the brick, of equal volume and of such size and number as to offer equal resistance to air flow. The dimensions of the capillaries are then computed. The method is limited to materials having open pores. F. SALT.

Vapour absorption of a fired earthenware body. C. L. DEEDS (J. Amer. Ceram. Soc., 1928, 11, 769—770).—Three specimens of an earthenware body, fired to different temperatures, were placed over 10% sulphuric acid for 36 days and the increase in weight was noted at intervals. Within limits, the amount of vapour absorption was proportional to the porosity, but the most porous specimen (20.25% absorption) showed a higher rate of vapour absorption and also greater variation. F. SALT.

Silica dust in lungs. HEFFERNAN and GREEN.—See XXIII.

PATENTS.

Heat-resistant borosilicate glass. L. HOCHSTEIN, Assr. to E. T. BROWN (U.S.P. 1,676,331, 10.7.28. Appl., 10.4.25. Ger., 15.3.25).—The glass consists of silica, boric oxide, alumina, and zirconia; its coefficient of expansion is about 0.0000017. H. ROYAL-DAWSON.

Manufacture of abrasive articles. D. E. WEBSTER, Assr. to NORTON Co. (U.S.P. 1,681,891, 21.8.28. Appl., 26.10.26).—Abrasive material and a vulcanising agent are added to rubber latex (30% or over), the resulting paste being shaped as desired, dried, and vulcanised. C. HOLLINS.

Manufacture of glass, sand, or flint paper, emery cloth, and like abrasives. C. A. KLEIN and R. S. BROWN (U.S.P. 1,687,453—4, 9.10.28. Appl., 28.10.25. U.K., 26.8.25).—See B.P. 258,412 and 260,704; B., 1926, 948; 1927, 44.

Glass furnaces. UNITED GLASS BOTTLE MANUFACTURERS, LTD., and T. C. MOORSHEAD (B.P. 297,856, 1.6.27).

Glass-annealing lehrs. BRIT. HARTFORD-FAIRMONT SYND., LTD., T. WARDLEY, and H. V. E. M. RENN (B.P. 297,865, 29.6.27).

IX.—BUILDING MATERIALS.

Road-surfacing materials. W. J. A. BUTTERFIELD (J.S.C.I., 1928, 47, 293—309r).—A review of the development of bituminous materials for surfacing roads in non-urban areas. Bitumen, according to the definition adopted in June, 1928, by a Committee instituted by the International Road Congress of 1926, comprises:—"Mixtures of natural and pyrogenous hydrocarbons and of their non-metallic derivatives, which may be gaseous, liquid, viscous, or solid, but must be completely soluble in carbon disulphide." Road tar is thus recognised as containing a large proportion of bitumen, and when it is necessary to distinguish the bitumen of asphalt from that in tar the former must, in future, be termed "asphaltic bitumen." The provisions of successive standard specifications for tar for surface dressing, and for tar for making tar macadam, are given. Recent specifications increase the consistency of the tar used for both purposes. Methods of testing the consistency of road tars are described, and comparative figures given for the Hutchinson viscosimeter and the modified Redwood instrument adopted by the British Road Tar

Association. The former requires 15 to 20 times as much tar as the latter for a test, and is otherwise less convenient in use. Good road tars are characterised by a moderate content of "free carbon" and of high-boiling viscous oils, as well as of naphthalene and phenols, both of which are indicative of the relative stability of other components. Tar has constituents which correspond to the vehicle of paint, which cause it to set and dry, whereas asphaltic bitumen ordinarily is wholly stable, and the necessary fluidity for application is secured solely by heating it. Both tar and asphaltic bitumen are, however, often made up into emulsions for ease of application. The stability of the constituents of asphaltic bitumen delays the ultimate disintegration of its coating as compared with tar, and efforts are now directed to rendering tar more nearly asphaltic constitutionally. Some, but not all, tars blend with asphaltic bitumen, and the resultant mixtures combine good features of both materials. Blinding or gritting materials need careful selection and screening in order that they shall both prevent the bituminous material adhering to tyres, and preclude slipperiness, whether the road surface is dry, damp, or wet. Fuller investigation of the action of dust and mud from different gritting materials, as dry and wet lubricants, is called for. The effect of the dust and mud resulting from the wear of bituminous surfaces is discussed in relation to roadside vegetation and rain washings. The silt conveyed by the latter is detrimental to fish life, owing to the fineness and colloidal character of the particles. The toxicity to fish of solutions of many tar products is set out for different months of the year. Trout are killed in 1 hr. by solutions of 1 pt. (in August) to 5 pts. (in December) of phenol or mixed cresols per 100,000. The silt from bituminous surfaces may be dangerous to fish even if it is free from phenols or other distinctive tar products. A comparison is made of three trials in which the whole of the rain washings from trafficked roadways were passed for about 6 months into small ponds containing trout. The surfacing materials were:—(1) a thin tar, with a large content of naphthalene, on granite; (2) an asphaltic bitumen on old tarred macadam made up with fresh granite, and (3) "Tarvia" (mainly coal tar of high consistency) on granite. No trout were killed at any time by the washings from (2) or (3), and from (1) only a few days after application, and after the lapse of 4 months, when the coating had been broken up by wear. In the absence of special channels for the collection and delivery of the road washings into a small pond or stream, the danger to fish from tar or other bituminous road surfacings is stated to be remote, and to be altogether negligible if the washings are aerated or filtered by passage over pasture or other land. Cement surfaces, both bare and dressed with tar or asphaltic bitumen, are briefly discussed.

Analysis of portland cement for factory purposes. W. J. PITT (Chem. Eng. and Min. Rev., 1928, 20, 402—406).—The method of analysis follows very closely the ordinary procedure of that of silicates. Full manipulative details are given for determination of silica, alumina, ferric oxide, calcium oxide, magnesium oxide, sulphuric anhydride, alkalis, and insoluble matter. C. A. KING.

PATENTS.

Curing cementing materials. BARBER ASPHALT Co., Assees. of F. W. McRAE (B.P. 291,784, 23.4.28. U.S., 10.6.27).—Concrete etc. after it has dried on the surface but before it has set, *i.e.*, after about 24 hrs., is sprayed or painted with a waterproof coating consisting, *e.g.*, of a solution of asphaltic material in a petroleum distillate, *d* 1.44—1.56. L. A. COLES.

Apparatus for making a spumous mass of cementitious material. G. B. HINTON (B.P. 297,638, 27.2.28).—Apparatus for carrying-out the process of U.S.P. 1,657,716 (B., 1928, 266) is described.

F. G. CROSSE.

Manufacture of cellular cementitious materials. G. B. HINTON (U.S.P. 1,687,067, 9.10.28. Appl., 2.2.28).—See B.P. 297,638; preceding.

Floor covering (B.P. 275,949).—See XIII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Mechanism of the blast furnace [smelting of iron ores] with reference to sintering and the precipitation of carbon. B. OSANN (Arch. Eisenhüttenw., 1928—9, 2, 137—143; Stahl u. Eisen, 1928, 48, 1402—1403).—By smelting a mixture of small pieces of iron ore, marble, and coke in a crucible in a coke fire, and blowing a blast of air through the mixture the mechanism of the blast-furnace smelting of iron ores may be followed if the process be interrupted after varying periods. In this way it has been shown that, in contact with iron ore, carbon monoxide decomposes at 300° and the carbon liberated is deposited in a finely-divided state in the pores of the ore, thus causing progressive breaking up of the ore lumps. This is followed by the reduction of the iron oxide by the carbon on the surface; in the hot zone of the blast furnace this reaction takes place simultaneously with the deposition of the carbon and thus produces a highly carburised iron, whereas in the cooler zones the carbon dust tends to accumulate faster than it is used up. The reduction process is accompanied by sintering of the ore particles to the fluxes and, if the correct quantity of lime is not present, this is followed by fusion caused by ferrous oxide entering the slag. The ferruginous slag then acts as a decarburising agent on the iron first formed. The carburisation of the iron in the blast furnace is dependent on the fineness of the charge, too many fines hindering the diffusion of gases and thereby reducing the amount of carbon deposited. Many other phenomena observed in blast-furnace working may be explained by the results obtained by the crucible method outlined above.

A. R. POWELL.

Coke-oven gas as a fuel for the Siemens-Martin furnace. G. BULLE (Stahl u. Eisen, 1928, 48, 1353—1362).—A summarised review of modern practice in Germany describing the methods employed in operating open-hearth steel furnaces with coke-oven gas and with mixtures of this gas with flue gases and with producer and flue gases. The composition and calorific

value of the gas mixtures, the effect of pre-heating, and the types of gas burners used are briefly discussed.

A. R. POWELL.

Effect of cold-rolling and annealing at different temperatures on the tensile properties and structure of high-grade soft iron sheets. E. MARKE (Arch. Eisenhüttenw., 1928—9, 2, 177—184; Stahl u. Eisen, 1928, 48, 1404—1405).—If soft iron sheets are annealed at 650—850° after cold-rolling to reduce the cross-sectional area by 5—20% a coarsely crystalline structure is obtained, but at 900° complete recrystallisation into a fine-grained structure is obtained. If this operation is carried out in annealing boxes, however, the sheets are liable to adhere to one another. With a reduction in area of less than 5% or more than 20%, annealing at 650—930° relieves the strain induced by cold-working without appreciably affecting the mechanical properties, but, for obtaining a very fine-grained structure, a prolonged anneal at 580° is recommended after a reduction in area of more than 20% by cold-rolling.

A. R. POWELL.

Evaluation of case-hardening materials [for iron]. F. HEBLER (Chem.-Ztg., 1928, 52, 775—776).—For temporary case-hardening of iron, no better results are obtained with wood charcoal than with other carbonaceous materials, such as coke, lignite, etc. The degree of case-hardening is scarcely affected by the addition of chalk to the carbon, but a progressive increase in the thickness of the hardened layer is obtained by the use of the carbonates of sodium, strontium, and barium.

F. R. ENNOS.

Causes of failure of wrought iron chain and cable. H. J. GOUGH and A. J. MURPHY (Proc. Inst. Mech. Eng., 1928, 293—326).—Apart from obvious causes, failure of wrought iron chain may be due to overheating or burning the metal during manufacture or to progressive deterioration of the weld by reason of service shocks and strains. An important cause of brittleness is associated with the use of chain, *e.g.*, snatch loading, rattling through hawse pipes, cleaning in rumbling machines, which destroys the ductility of the surface layer and results in cracking in later use. The customary reannealing of iron chains effectually restores the ductility.

C. A. KING.

Practical corrosion research [applied to iron and steel]. E. H. SCHULZ (Stahl u. Eisen, 1928, 48, 1393—1401).—A review of recent work on the corrosion of iron and steel and on the development of corrosion-resisting steels in which the action of various corroding media is discussed, together with the influence of copper and silicon in reducing the rate of corrosion of constructional steels.

A. R. POWELL.

Flocks in chromium-steel. W. AICHHOLZER (Stahl u. Eisen, 1928, 48, 1332—1334).—Steels containing 0.8—1.2% C and 1—3% Cr frequently contain glistening nodular "flocks" in the inside of large ingots. Microscopic and macroscopic examination shows the existence of hair cracks between the surfaces of the flocks and the remainder of the ingot. The microstructure of the steel round these cracks reveals the presence of a carbide-rich constituent containing about 1.5—2% C and 13%

Cr, as shown by its behaviour during heat treatment. This constituent suffers only a very small increase in volume during hardening, whereas the remainder of the ingot increases 0.13% in length during this treatment. The strains set up, therefore, by heat treatment produce hair-cracks along the zones in which the harder constituent has segregated, and the characteristic bright crystalline appearance of the surface of these cracks gives rise to these flocks. A. R. POWELL.

Electrochemical behaviour and tendency to rusting of chromium steels. O. MEYER and K. ROESCH (Arch. Eisenhüttenw., 1928—9, 2, 121—127; Stahl u. Eisen, 1928, 48, 1372—1373).—Measurements of the potential of a 13% chromium steel in various salt solutions, together with corrosion tests, show that rusting commences whenever the potential falls below the value $\epsilon_R = -0.015$ to -0.010 volt. In acid solutions the potential of chromium steels becomes more positive and the rate of corrosion decreases with an increase in the quenching temperature. The higher the carbon content of the steel the higher is the temperature from which the steel must be quenched in order to prevent rusting, as the more nearly the structure approaches a single solid solution the greater is the chemical resistance; in this sense, therefore, carbon must be considered as an objectionable impurity in rust-resisting steels. Other things being equal a highly polished surface resists corroding media to a greater extent than does a rough surface, and the difference between the behaviour of rough and smooth surfaces is more marked in hardened steel than in forged or annealed steel.

A. R. POWELL.

Determination of vanadium in steel.—F. IBBOTSON (Analyst, 1928, 53, 531).—Evans and Clarke's method (B., 1928, 786) for the determination of vanadium was found very satisfactory for an alloy steel containing also chromium, molybdenum, tungsten, nickel, and cobalt, but the glyoxime precipitate formed during the removal of nickel was found untractable, and for a steel containing about 0.5% each of nickel and vanadium, together with other metals, the nickel was not removed and the filtration from the two ferrocyanides was less difficult than from vanadium ferrocyanide alone. Two Munktell 15 cm. filters on a ribbed funnel were used by the author instead of a pulp filter. D. G. HEWER.

Citric acid solubility and hardness of basic slag.

H. WEISS (Arch. Eisenhüttenw., 1928—9, 2, 81—85; Stahl u. Eisen, 1928, 48, 1403—1404).—The percentage of phosphoric acid soluble in citric acid in basic slag increases with the silicification factor to a maximum between 0.42 and 0.45. For pig iron low in silicon the necessary silica to obtain this factor should be added in the form of sand to the converter $\frac{1}{2}$ —1 min. before the end of the blow. Slags with a low lime content and a high content of phosphoric acid often have a good citric acid solubility in spite of a low silicification factor owing to the high temperature required in the converter and consequent greater fluidity of the slag. On the other hand very basic slags, e.g., with 60% of lime, having a high silicification factor yield only 85—90% of their phosphoric acid to citric acid owing to their greater viscosity when molten. The citric acid solubility is

greater with soft slags than with hard slags that contain a large percentage of ferrous or manganous oxides which, by combining with the silica, reduce the amount available for the formation of the soluble silicophosphate.

A. R. POWELL.

Examination of the structure of slags. O. GLASER (Arch. Eisenhüttenw., 1928—9, 2, 73—79; Stahl u. Eisen, 1928, 48, 1373—1374).—A review of the various physical, optical, and thermochemical methods used in examining the constitution and structure of slags, with especial reference to those produced in smelting iron ores and in the manufacture of steel. A. R. POWELL.

Manufacture of copper castings with a high electrical conductivity. G. MASING and C. HAASE (Wiss. Veröff. Siemens-Konz., 1928, 7, 321—334).—The electrical conductivity and the density of sand castings of copper have been determined after deoxidation with phosphorus, lithium, beryllium, magnesium, calcium, aluminium, silicon, and boron suboxide. The use of phosphorus as a deoxidiser results in a gradual increase in the density as the amount of phosphorus retained by the casting increases, but the electrical conductivity is rapidly decreased. Lithium, added in the form of a 10% alloy with copper, effects complete removal of oxygen and sulphur from copper; 0.02% Li in copper ensures sound, dense castings of very high sp. gr. and electrical conductivity, but the melting must be carried out under a flux of soda-glass to obtain a smooth surface to the casting free from the corrugations which are otherwise obtained. Less than 0.04% of beryllium added to molten copper efficiently removes oxygen, but sulphur is retained in the form of minute thread-like inclusions, which, however, do not affect the density, conductivity, or mechanical properties. Magnesium reduces cuprous oxide and sulphide to metal, but the magnesium compounds formed are not readily removed from the metal, so that the surface of the castings shows irregularities due to nests of oxide inclusions; the density and conductivity of the metal are, however, satisfactory. Similar results are obtained with calcium and aluminium as deoxidisers, whereas silicon tends to lower the conductivity below the standard value. Efficient deoxidation is obtained with boron suboxide only when the metal is heated at 1300°; simple heating at 1400° without a deoxidiser also yields castings of good quality, owing to the rapidity with which occluded gas is expelled at this temperature, and to the interaction of sulphide and oxide inclusions. The good results obtained by the use of beryllium for producing high-conductivity copper castings are easily reproducible on a large scale using 0.3 g./kg. of beryllium in the form of a 10% alloy with copper. A. R. POWELL.

Anomalies of annealing after cold-beating of copper and brasses. EUGÈNE (Compt. rend., 1928, 187, 378—380).—The beginning and end of the zone of germination of the annealing of pure copper are marked by two anomalies on the temperature-hardness curves, the latter being the more marked, and corresponding with the most favourable annealing temperature for working copper. The addition of zinc increases and diminishes the first and second anomalies, respectively. The anomalies obey the law of displaced equilibria,

although they accompany an irreversible change of state in which there is no thermal reaction.

J. GRANT.

Condenser materials and blue powder in zinc smelting. R. W. MILLAR (Min. and Met., 1928, 9, 395).—At 850° mixtures of zinc vapour and carbon monoxide react very slowly in a silica tube, whereas from thermodynamic considerations reaction should take place according to the equation: $\text{Zn (gas)} + \text{CO} = \text{ZnO} + \text{C}$. The rate of reaction is not affected by the presence of zinc oxide, alumina, or clay from which the iron oxide has been removed; ferruginous clay is, however, an active catalyst as it catalyses the reaction $2\text{CO} = \text{CO}_2 + \text{C}$, and the carbon dioxide formed then rapidly oxidises the zinc vapour. It is suggested, therefore, that the presence of iron oxide in the clay from which condensers are made may be the cause or one of the causes of the formation of blue powder in zinc smelting.

A. R. POWELL.

Assay of low-grade tin ores and tailings. G. E. GABRIEL (Chem. Eng. and Min. Rev., 1928, 20, 407).—1 g. of the finely-ground sample is added to a cooled melt of 8 g. of sodium hydroxide and a small quantity of charcoal. If sulphides are present, the ore is first oxidised with aqua regia. After fusion, the cooled cake is dissolved in 50 c.c. of water and 50 c.c. of hydrochloric acid and transferred to a conical flask fitted with a stopper and tube which dips into a solution of sodium carbonate. Reduction to stannous chloride is effected by means of powdered antimony in an atmosphere of carbon dioxide and, while cooling, sodium carbonate is drawn into the flask to maintain a neutral atmosphere. The stannous chloride is titrated cold with iodine solution.

C. A. KING.

Electrodeposition of aluminium. D. B. KEYES, S. SWANN, JUN., W. KLABUNDE, and S. T. SCHICKTANZ (Ind. Eng. Chem., 1928, 20, 1068—1069).—An adherent coating of aluminium was obtained on a copper cathode by the electrolysis of the reaction products of aluminium and ethyl iodide (cf. Grignard and Jenkins, A., 1924, i, 951) treated with dry ether. A less successful result was obtained with tetraethylammonium bromide mixed with aluminium bromide. Aluminium electroplate would find application in economiser tubes, gas cylinders, cracking stills, etc.

C. IRWIN.

Influence of composition and cold-working on corrosion and increase of grain size of aluminium. L. GUILLET and BALLAY (Compt. rend., 1928, 187, 585—587).—Pure aluminium corrodes less than if only 98·81% pure in aqueous hydrochloric acid; pure nitric acid, and 3% sodium chloride solution. Both metals behave alike in 5% nitric acid and sulphuric acid solutions. The influence of cold-working is much greater in the case of the impure aluminium, both in increasing the corrosion and enlarging the grain size.

C. W. GIBBY.

The Madsenell process [for the degasification of metal surfaces before plating]. F. M. DORSEY (Ind. Eng. Chem., 1928, 20, 1094—1099).—The work is transferred along a line of tanks by an overhead crane. Oil is removed in a bath of potassium hydroxide solution with whale oil soap, working with a 6-volt

direct current. The article is rinsed, pickled, again rinsed, and is then ready for degasification. This is carried out in 95% sulphuric acid with a 12 volts *E.M.F.* The current rises to 50 amp. and then falls to zero when gas ceases to be evolved. Embedded oil and grease are removed as well as occluded gases. The preparation of a nickel plating bath and its control by means of bromothymol-blue are described. After 48 hrs. the bath must be filtered and its composition adjusted. Polarisation is minimised by working as near the neutral point as possible (p_{H} 4—6), by addition of hydrogen peroxide, and by superimposing an alternating current. A continuous plant for the treatment of wire etc. is described. The product is characterised by perfect adherence in any thickness, a maximum corrosion resistance, and a bond which allows of bending, welding, soldering, etc.

C. IRWIN.

Determination of small quantities of carbonate. VERNON and WHITBY.—See VII.

PATENTS.

Treatment of sulphide ores or sulphidic metallurgical products. F. KRUPP GRUSONWERK A.-G. (B.P. 297,597, 24.11.27. Addn. to B.P. 234,826; B., 1925, 676).—The material, contained in, *e.g.*, a rotary tubular furnace, is first roasted in an oxidising atmosphere which may contain air, in order to remove a portion only of the sulphur as sulphur dioxide. The product, mixed with reducing and sulphur-binding agents, such as alkaline-earth compounds, is then subjected to a combined reducing, roasting, and distilling treatment in a volatilisation operation so as to liberate a distillation product which is made to react with the gases in the atmosphere of the preliminary roasting zone or of the volatilisation zone to form metallic sulphates. The zinc sulphate so formed is extracted by lixiviation, any lead sulphate associated with it remaining behind as an insoluble residue.

M. E. NOTTAGE.

Treatment of ores. Q. BENT, E. BARNHART, and F. W. WOOD, Assrs. to BETHLEHEM STEEL Co. (U.S.P. 1,684,006, 11.9.28. Appl., 4.10.20).—In their passage through a moderately heated, slightly inclined, horizontal, rotary kiln having longitudinal ribs, wet plastic ores are partially dried and formed into masses of convenient size and in a condition suitable for subsequent furnacing.

F. G. CLARKE.

Purification of pig iron. F. WÜST (B.P. 274,438, 17.6.27. Ger., 17.7.26).—Molten pig iron is brought into contact with a dephosphorising and desulphurising slag in a rotary furnace, and on saturation of the slag the metal is purified finally by the introduction of a further quantity of fresh slag, which is afterwards used for the preliminary treatment of a later quantity of iron direct from the blast furnace.

C. A. KING.

Wrought articles of iron-chromium-nickel alloy. H. E. POTTS. From ELECTRO METALLURGICAL Co. (B.P. 297,045, 9.6.27).—An alloy of iron containing: 15—40% Cr, 2—15% Ni, 0·7—3% Si, 0·7—3% Mn, and 1% C, or less, is rolled into plates and formed into articles by welding, the articles having a high resistance to corrosion. [Stat. ref.]

C. A. KING.

Ferrous alloy. P. A. E. ARMSTRONG (U.S.P. 1,687,486, 16.10.28. Appl., 14.8.25).—The alloy contains approx. 9% Cr, 3.5% Si, 3% Ta, and 0.5% C. F. G. CROSSE.

Alloy. G. H. BENDER (U.S.P. 1,685,975, 2.10.28. Appl., 4.4.27).—Copper (70.3 pts.) is alloyed with tin (24.53 pts.) and lead (5.2 pts.). F. G. CROSSE.

Heat-treating copper-nickel-beryllium alloys. M. G. CORSON (B.P. 279,425, 3.10.27. U.S., 20.10.26).—An alloy containing copper, 0.1—2% (0.3%) Be, and up to 40% (4%) Ni is worked, heated to 900°, and either cooled slowly or quenched and reheated to 350—700°. [Stat. ref.] C. A. KING.

Alloy and its manufacture. A. L. FEILD, Assr. to ELECTRO METALLURGICAL CO. (U.S.P. 1,684,696, 18.9.28. Appl., 25.10.24).—In order to combine zirconium with copper, an alloy of aluminium and zirconium is introduced into a superheated bath of copper. F. G. CROSSE.

[Nickel] alloy. R. FRANKS and B. E. FIELD, Assrs. to HAYNES STELLITE CO. (U.S.P. 1,684,131, 11.9.28. Appl., 5.3.26).—The alloy claimed in U.S.P. 1,675,798 (B., 1928, 609) is modified by substituting titanium for zirconium. F. G. CROSSE.

[Silver] alloys resistant to chemical action. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,165, 30.6.27).—Silver alloys resistant to tarnishing by sulphides and to corrosion by hydrochloric acid contain at least 78% Ag and up to 22% of a mixture of aluminium with one or more of the elements thallium, magnesium, zinc, arsenic, antimony, bismuth, tin, or silicon. The alloy containing 78% Ag, 5% Al, and 17% Tl is claimed to resist almost completely the action of concentrated hydrochloric acid and to be suitable for the manufacture of chemical apparatus for handling this acid. A. R. POWELL.

Recovery of tin from tin scrap or cuttings. T. TWYNAM (B.P. 297,528, 11.7.27).—Tin scrap is immersed in commercial hydrochloric acid diluted with 3—4 times its volume of water, or in the acid liquor from galvanising pickling operations, the temperature being slowly raised to 80—90° so as preferentially to dissolve the tin, which is subsequently precipitated from solution by means of zinc. M. E. NOTTAGE.

Fluxes for use in melting aluminium or other non-ferrous metals. G. W. WARDLE (B.P. 297,635, 17.2.28).—Cryolite is crushed, fused at a temperature considerably above the m.p. of aluminium, and formed into blocks, which will float on the surface of the molten metal. M. E. NOTTAGE.

Polishing of chromium. METALS PROTECTION CORP., Asses. of J. C. PATTEN (B.P. 277,296, 24.6.27. U.S. 9.9.26).—Finely-divided, partly calcined hydrated alumina or bauxite, made by heating precipitated aluminium hydroxide at 600—1000°, is used either alone or mixed with stearic acid, a mixture of stearic acid and beeswax, or with jellied soap. M. E. NOTTAGE.

Manufacture of magnesium[chromium] alloy. W. R. VEAZEY, Assr. to DOW CHEM. CO. (U.S.P. 1,685,653, 25.9.28. Appl., 19.12.24).—Magnesium is alloyed with 0.01—0.1% of chromium. F. G. CROSSE.

Making synthetic pig iron. E. EDWIN, Assr. to A./S. NORSK STAAL (ELEKTRISK-GAS-REDUKTION) (U.S.P. 1,686,075, 2.10.28. Appl., 25.1.27. Norw., 10.12.25).—See B.P. 286,067; B., 1928, 372.

Production of wrought iron with increased tensile properties. G. G. GEDDA (U.S.P. 1,685,602, 25.9.28. Appl., 9.12.25. Swed., 17.7.25).—See B.P. 260,789; B., 1927, 46.

Refining of lead bullion containing other metals. G. K. WILLIAMS (U.S.P. 1,687,187—8, 9.10.28. Appl., 9.2.27. Austral., 6.3.26).—See B.P. 267,104; B., 1928, 270.

Producing metals or alloys low in carbon directly from ore etc. Direct-reduction process for producing carbon-binding metals or alloys. H. G. FLODIN and E. G. T. GUSTAFSSON, Assrs. [B] to H. G. E. CORNELIUS (U.S.P. 1,686,206—7, 2.10.28. Appl., [A] 17.12.24, [B] 7.4.25. Swed., [A] 12.1.24, [B] 27.11.24).—See B.P. 227,435 and 243,743; B., 1925, 766; 1927, 337.

Manufacture of sand mould for casting magnesium. G. MICHEL (U.S.P. 1,685,553, 25.9.28. Appl., 5.2.25. Fr., 4.10.24).—See B.P. 254,159; B., 1926, 755.

Impregnation of metal layers. N. MEURER, Assr. to METALLOGEN GES.M.B.H. (U.S.P. 1,688,127, 16.10.28. Appl., 20.12.22. Ger., 28.1.22).—See B.P. 211,181; B., 1924, 428.

Alloy and its application to the manufacture of electrical conductors. W. S. SMITH, H. J. GARNETT, and J. A. HOLDEN (U.S.P. 1,688,247, 16.10.28. Appl., 6.1.27. U.K., 10.7.26).—See B.P. 278,454; B., 1927, 912.

Electrolytically separating alloys of silver with other precious or base metals. R. CARL (U.S.P. 1,687,056, 9.10.28. Appl., 27.7.23. Austr., 24.11.22).—See G.P. 377,144; B., 1923, 936 A.

Placer mining by direct leaching with cyanide or other solvents. B. STOCES (B.P. 296,813, 9.6.27).

Process and apparatus for welding. W. E. BEATTY. From BELL TELEPHONE LABS., INC. (B.P. 297,493, 23.6.27).

XI.—ELECTROTECHNICS.

Apparatus for electrolysis with high current densities. E. DUHME and H. GERDIEN (Wiss. Veröff. Siemens-Konz., 1928, 7, 304—306).—The apparatus comprises a lower electrode consisting of a solid copper cylinder inside a glass mantle covered with a quartz plate, and an upper electrode consisting of a smaller, hollow, cylindrical electrode of platinum or other suitable metal. The copper cylinder has a vertical hole through the middle lined internally with a platinum tube, cone-shaped at its upper end, which terminates 2 m. above the copper and is directly below a hole in the centre of the quartz plate. The liquid to be electrolysed is passed downwards through a vertical tube in the centre of the upper electrode directly on to the hole in the lower electrode, so that 90% passes through the hole and 10% circulates back to the upper electrode.

A. R. POWELL.

Electrochemical behaviour of chromium steel. MEYER and ROESCH. **Copper castings.** MASING and HAASE. **Electrodeposition of aluminium.** KEYES and others. **Madsenell process.** DORSEY.—See X. **Conductivity of soil extracts.** SEN.—See XVI. **Determining the p_H values of beers etc.** STOCKHAUSEN and ROTHENBACH.—See XVIII.

PATENTS.

Dry cell. L. MELLERSH-JACKSON. From C. F. BURGESS LABS., INC. (B.P. 297,400, 13.6.27).—A perforated anode in the form of a shallow pan is provided with gelatinisable electrolyte on one side and an expansion chamber on the other. Opposed electrodes are spaced apart by a collar of fibrous material impregnated with polymerised tung oil. J. S. G. THOMAS.

Manufacture of accumulator plates. I. G. FARBENIND. A.-G. (B.P. 290,193, 2.4.28. Ger., 9.5.27. Addn. to B.P. 284,352; B., 1928, 717).—Prisms of lead alloy are inserted or pressed into cells in a lead skeleton structure, and the whole is pressed to form a massive block or plate, from which the metal alloyed with the lead is subsequently removed. J. S. G. THOMAS.

[Positive plates for] alkaline storage batteries. J. F. MONNOT (B.P. 297,996, 4.11.27).—A perforated metallic tube, *e.g.*, of steel, of substantially circular cross-section is filled with alternate layers of perforated nickel foil and nickel hydroxide, the foil employed being greater in size than the internal cross-sectional area of the tube so that it makes good contact with the wall of the tube. J. S. G. THOMAS.

Battery composition. P. J. KELLEHER (U.S.P. 1,685,674, 25.9.28. Appl., 16.3.26).—A composition capable of preventing "sulphation" in storage batteries consists of 84% of magnesium sulphate, 3% of ammonium sulphate, 6% of potassium sulphate, and 7% of aluminium sulphate. F. G. CROSSE.

Electrolyte for storage batteries. A. B. WERBY, Assr. to AMER. AUTOMOTIVE CORP. (U.S.P. 1,684,852, 18.9.28. Appl., 5.6.28).—Dilute sulphuric acid containing ammonium acetate is used. F. C. CROSSE.

Electrolyte for lead accumulators. M. KUGEL (B.P. 280,197, 28.10.27. Ger., 6.11.26).—Sulphation in lead accumulators is minimised by adding a small quantity (0–20 g./litre) of phosphoric acid to the dilute sulphuric acid usually present. H. ROYAL-DAWSON.

Electrolyte for accumulators. M. KUGEL (B.P. 291,020, 28.10.27. Ger., 24.5.27. Addn. to B.P. 280,197; B., 1928, preceding).—Sulphation is avoided by adding fresh phosphoric acid from time to time to the electrolyte to replace loss due to wastage.

H. ROYAL-DAWSON.

Anodes for gas-discharge vessels. SIEMENS & HALSKE A.-G. (B.P. 279,501, 22.10.27. Ger., 23.10.26).—The anode is made of conducting refractory material, *e.g.*, tantalum or molybdenum, which absorbs gases at its normal working temperature. J. S. G. THOMAS.

Electrolytic rectifier. W. C. READ, Assr. to ELECTRO METALLURGICAL Co. (U.S.P. 1,684,684, 18.9.28. Appl., 8.3.27).—In a rectifier consisting of an anode of lead composition and an electrolyte of 10–40% sulphuric

acid containing the sulphate of a metal, the cathode is composed of silicon alloyed with 10–40% of a metal of the titanium group. F. G. CROSSE.

Measuring the intensity of radiation especially of sources of ultra-violet light. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 296,198, 8.9.27).—The intensity of ultra-violet radiation of 210–350 μ is determined by exposing solutions of phototropic compounds, *e.g.*, leucocyanines, carbinols, or sulphurous compounds of crystal-violet, malachite-green, fuchsin, etc., containing small quantities of substances counteracting the photochemical colour change, *e.g.*, potassium cyanide, potassium hydroxide, and sulphurous acid, respectively, to the action of the rays, and comparing with standard solutions of the dyes themselves the intensity of the coloration produced.

L. A. COLES.

Self-baking electrodes [for electric furnaces]. C. W. SÖDERBERG, Assr. to NORSKE A./S. FOR ELEKTROKEM. IND. (U.S.P. 1,686,474, 2.10.28. Appl., 18.9.26. Norw., 19.9.25).—See B.P. 258,560; B., 1927, 257.

Electric battery of the Leclanché type. G. N. ANTONOFF, Assr. to M. A. ADAM (U.S.P. 1,687,051, 9.10.28. Appl., 26.5.24. U.K., 8.6.23).—See B.P. 225,889; B., 1925, 105.

[Electrodes for] batteries, electric accumulators, and electrolysing apparatus. R. OPPENHEIM, Assr. to SOC. ANON. LE CARBONE (U.S.P. 1,687,307, 9.10.28. Appl., 30.9.25. Fr., 10.12.24).—See B.P. 244,417; B., 1927, 17.

Sludging of transformer oils (B.P. 278,365).—See II.

XII.—FATS; OILS; WAXES.

Fatty acids of Egyptian butter fats. H. ATKINSON (Analyst, 1928, 53, 520–530).—An analytical examination of the butter of the gamoos, cow, goat, and Syrian samn shows that the usual methods are insufficient to determine adulteration when the origin of the butter is unknown. The variations in the Polenske and Reichert-Meissl values allow of additions of appreciable proportions of coconut or palm-kernel oils, so that the methods of Kirschner, Blichfeldt, Ewers, Cassal, and Gerrans and Gilmour fail. Since the acids differentiating the fats of sheep and goats from those of cows and gamoos (octoic and decaoic) are soluble in aqueous alcohol, Shrewsbury and Knapp's method is of little use. If, however, 1 g. of saponified fat is distilled with 210 c.c. of water, the titration value of the non-volatile acids expressed as mg. of potassium hydroxide obtained by subtracting that of the volatile acids from the saponification value is, in general, lower in the sheep and goat butters. Further, the sum of oleic acid and acids insoluble in 62% aqueous alcohol, subtracted from the total non-volatile acids, is, within reasonable limits, a constant for each fat, and this constant is increased by coconut or palm-kernel oil practically in proportion to the amount present, and the residual acids are increased by about 12 units for every 10% of fat. These latter are decreased by beef fat by about 2 units for every 10% of fat. Having thus ascertained the purity of a butter fat,

the origin may be deduced from the Polenske-Reichert-Meissl ratio or the "insoluble silver" value. Tables of figures for various types of butter from cows, gamoos, goat, and Syrian samn are given, both pure and with addition of varying proportions of palm-kernel oil, coconut oil, and beef fat, also a chart comparing the acids of Egyptian butter fats with those of coconut and palm-kernel oils.

D. G. HEWER.

Duclaux method for determination of volatile fatty acids, and its application to the determination of butter fat in margarine. A. KNETEMANN (Rec. trav. chim., 1928, 47, 950—970).—In the Duclaux method for the determination of volatile fatty acids (A., 1896, ii, 504), the relation between the concentration of the acid in the vapour and the liquid (distillation value = C) is constant at each stage of the distillation and varies with the acid used. This value has been determined experimentally for formic, acetic, propionic, butyric, valeric, hexoic, and octoic acids, and it increases regularly with increase of the carbon chain. Distillation curves are plotted for the above acids and for benzoic and salicylic acids. The constants deduced by Duclaux and by Boekhout and de Vries (A., 1917, ii, 50) are shown to be erroneous. For the determination of butter fat in margarine the fat is hydrolysed with concentrated potassium hydroxide solution in presence of glycerol at 135° (higher temperatures must be avoided as volatile acids are produced), and after dilution and acidification the solution is extracted with light petroleum. The filtered aqueous solution is distilled and two consecutive portions (100 and 200 c.c.) of the distillate are titrated with standard alkali. Substitution of these titration values in a formula deduced experimentally from the results obtained using artificial mixtures of butter fat, oleo oil, and coconut oil, gives the percentage value. The results appear to be trustworthy even when abnormal coconut oil is present. H. BURTON.

Detection and determination of arachis oil in olive oil. E. JAFFE (Annali Chim. Appl., 1928, 18, 368—386).—The lithium soaps of arachis oil, either pure or mixed with olive oil, are precipitated from their alcoholic solution at a higher temperature than the corresponding soaps of pure olive oil. Moreover, the fatty acids obtained by decomposing the lithium salts collected at 17—18° are precipitated from their solution in 90% alcohol only if arachidic acid is present. The details of the procedure to be followed are given. From the results obtained the proportion of arachis oil present may be determined by means of a table giving various temperatures of precipitation of the lithium salts of the fatty acids (ranging from 24° to 57°) and temperatures of crystallisation of the crude arachidic acid from 90% alcohol (over the range 15—43°).

T. H. POPE.

Determination of oil in seeds, etc. F. GOGOLEV (Masloboino-Shirowoje Djelo, 1927, No. 5, 20—21).—It is recommended that seeds should be ground in a mortar, oil-cake in a mortar or mill, and kept for 12 hrs. in the solvent before extraction (5—6 hrs. extraction for seeds, 2—3 hrs. for oil-cake); if this is omitted, an extra hour's extraction must be given.

C. HOLLINS.

Margarine. VITOUX.—See XIX.

PATENTS.

Separation of butter fat from buttermilk and buttermilk whey. Recovering butter from buttermilk. A. L. RUSHTON, Assr. to M. H. LANE (U.S.P. 1,683,728—9, 11.9.28. Appl., [A] 16.2.27, [B] 29.9.27).—(A) Sour cream buttermilk is treated, e.g., by heating or adding an acid or alkali, so that the buttermilk having a heavy casein content settles from the clear whey. The latter is then passed slowly through a cream separator. (B) Buttermilk whey is passed through a separator at the usual rate for milk, the recovered "buttermilk cream" is then passed through the separator at a slower rate; the buttermilk cream is finally churned.

F. G. CLARKE.

Stabilisation of fats, oils and waxes. J. Y. HUBER, JUN. (U.S.P. 1,680,047, 7.8.28. Appl., 4.2.24).—0.01% of furfuraldehyde added to animal or vegetable oils or fats retards the development of rancidity.

C. HOLLINS.

Manufacture of an insoluble soap from aldehyde-fatty acid mixtures [catalytic oxidation products] of mineral oil. J. H. JAMES, Assr. to C. P. BYRNES (U.S.P. 1,681,237, 21.8.28. Appl., 7.3.19).—The mixture obtained by catalytic oxidation of petroleum hydrocarbons contains ω -aldehyde-derivatives of acids C_{12} to C_{15} , together with aldehydes and hydrocarbons. The former are converted into soaps by treating with caustic alkali and separate as gels.

C. HOLLINS.

Manufacture of sulphonic acids derived from non-aromatic carboxylic acids. I. G. FARBERIND, A.-G. (B.P. 288,612, 21.6.27. Ger., 21.6.26. Cf. B.P. 272,967; B., 1928, 703).—Non-aromatic carboxylic acids containing more than eight carbon atoms in the molecule are sulphonated by strong sulphonating agents (e.g., sulphur trioxide, chlorosulphonic acid) at above 35° (e.g., 100°). Neutral media, unacted on in the working conditions, e.g., carbon tetrachloride, and/or catalysts may be employed.

E. LEWKOWITSCH.

Manufacture of a finely-divided dry soap product. INDUSTRIAL SPRAY-DRYING CORP., Assees. of R. L. HOLLIDAY (B.P. 269,506, 21.3.27. U.S., 19.4.26).—See U.S.P. 1,621,506; B., 1927, 339.

Purification of oils and fats. W. GENSECKE, Assr. to AMER. LURGI CORP. (U.S.P. 1,685,195, 25.9.28. Appl., 5.2.25. Italy, 15.2.24).—See B.P. 229,283; B., 1925, 889.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Unreliability of visual inspection of exposure tests of paints. P. H. WALKER and E. F. HICKSON (Ind. Eng. Chem., 1928, 20, 997).—After three years' exposure of two coats of red lead paint on steel, visual inspection indicated an advanced state of decay of the coating, although no serious corrosion had occurred. On vigorous washing with soap and water the paint was found to be quite intact and in excellent condition. This was confirmed by the electrical conductivity method (B., 1928, 578). Deposits of dirt, especially on red lead paint, frequently have the appearance of iron rust.

S. S. WOOLF.

Analysis of Bordeaux spirits of turpentine by rise of temperature on mixing with sulphuric acid.

Mlle.) M. BARRAUD (Bull. Inst. Pin, 1928, 47, 73—76).—The rise of temperature observed when freshly prepared spirits of turpentine is mixed with sulphuric acid ($d^{15} 1.72$) is constant for products of the same origin, but increases rapidly with ageing of the turpentine. The first 20% of the distillate gives fairly constant values whether the sample is oxidised or not; hence the addition of 2.5% of white spirit can be detected. The rise of temperature for turpentines of different origins varied from 71.5° to 116.5°.

CHEMICAL ABSTRACTS.

Inhalation experiments with certain lacquer solvents. H. F. SMYTH and H. F. SMYTH, JUN. (J. Ind. Hygiene, 1928, 10, 261—271).—The toxicity of lacquer solvents, with the exception of benzene, has been studied by means of gassing tests on guinea-pigs; the concentrations employed were such as might be expected to occur in practice, depending on the volatility and percentage of the solvent in the lacquer. Ethyl and amyl acetate, and possibly butyl acetate, are usable with safety in the concentrations generally employed, and possibly in somewhat higher concentrations; gasoline, turpentine, and xylene are possibly harmful in concentrations materially greater than those in present use; the proportions of toluene and butyl alcohol now employed represent very nearly the upper limit of safety. With suitable exhaust ventilation these limits may be exceeded with impunity. F. R. ENNOS.

False equilibria, with special reference to rosin solutions and gold size. R. P. L. BRITTON (J. Oil and Colour Chem. Assoc., 1928, 11, 323—330).—A general discussion on "false equilibria" is illustrated by the behaviour of solutions of rosin in methylated spirit etc. and of gold size (considered as a combination of a varnish and a strong solution of drier). The author indicates the possibility of a mobile equilibrium between the super-fused and the crystalline varieties of varnish material, *e.g.*, gums, linoleates, etc. The use of protective agents or stabilisers to control such equilibria is of importance in the varnish industry, the superiority of turpentine over white (petroleum) spirit as a solvent being an example of this type of protection. S. S. WOOLF.

Rosin size. DELCROIX.—See V.

PATENTS.

Anti-corrosive heat-resisting paint or composition. J. C. CARROLL (B.P. 297,342, 18.6.27).—A powdered and dried titanium ore, *e.g.*, ilmenite, ground in raw linseed oil and thinned with dark boiled oil, varnish, and white (petroleum) spirit is claimed. S. S. WOOLF.

Fatty essence [oxidised turpentine oil]. L. DESALBRES and J. DUBOURG (F.P. 620,462, 19.8.26).—Turpentine etc. is oxidised with air or oxygen in presence of a catalyst. C. HOLLINS.

Production of white lead. F. T. BAILEY and W. AUSTIN (B.P. 273,287, 16.6.27. U.S. 22.6.26).—A mixture of dilute acetic acid and powdered lead oxide is repeatedly sprayed through a current of carbon dioxide in suitable apparatus until the oxide is completely converted into white lead. As hydrating agent or catalyst, ammonium nitrate, nitric acid, etc. may replace acetic acid. Conversion into white lead is direct, the intermediate formation of lead acetate being eliminated. S. S. WOOLF.

Manufacture of coloured compounds [lakes]. I. G. FARBENIND. A.-G. (B.P. 275,943, 21.6.27. Ger., 12.8.26).—Compounds which are not basic dyes and are free from acid groups, but contain amino-groups, are treated in acid solution with sodium phosphotungstomolybdate or other similar salt. Complex phosphotungstomolybdates of 4:4'-tetramethyldiamino-diphenylmethane, -benzophenone, -benzhydrol, and *p*-aminobenzaldehyde are described. C. HOLLINS.

Production of colouring materials. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,076, 14.3.27. Cf. B.P. 267,924; B., 1928, 708).—For the dispersion of colouring matters (*e.g.*, soot, umber) these are mixed with sulphonated mineral oil etc. as used in the prior patent for treatment of textiles. C. HOLLINS.

Manufacture of transparent lacquers. CHEM. FABR. DR. K. ALBERT G.M.B.H. (G.P. 448,297, 11.6.25).—The free acid of natural resins, with or without admixture with synthetic resins, is partly or wholly converted into lithium salts (for spirit lacquers) or cadmium salts (for oil lacquers). *E.g.*, molten French colophony is treated at 200—250° with 1% of cadmium oxide, which rapidly dissolves, and then with successive small amounts of calcium hydroxide: American colophony is melted with cadmium resinate and esterified with glycerol: a phenol-acetone-formaldehyde resin is melted with cadmium oleate and colophony. The products yield lacquers which remain transparent for long periods. C. HOLLINS.

Prevention of livering in pigmented carbohydrate-compound compositions [cellulose esters and ethers]. E. I. DU PONT DE NEMOURS & Co., Asses. of J. D. MCBURNEY and E. H. NOLLAU (B.P. 278,696, 26.9.27. U.S., 5.10.26).—The livering is prevented or removed by the addition of not more than 5% of a compound having a dissociation constant of 1.0—4.7, *e.g.*, citric, malic, or tartaric acid. L. A. COLES.

Manufacturing floor covering etc. ARMSTRONG CORK Co. (B.P. 275,949, 4.7.27. U.S., 13.8.26).—Hard floor-covering materials are surfaced with pigmented nitrocellulose lacquers applied in a block printing machine, the atmosphere around which is cooled and saturated with lacquer solvents, thus precluding evaporation of solvents from the lacquer. The surfaced material is subsequently dried in a curing chamber from which solvent vapours are withdrawn. S. S. WOOLF.

Preparation of derivatives of resinous phenol-aldehyde condensation products. I. G. FARBENIND. A.-G. (G.P. 449,276, 14.1.20. Addn. to G.P. 386,733; B., 1924, 604).—Fusible phenol-aldehyde resins are treated with halogenated aliphatic acids, *e.g.*, mono- or di-chloroacetic, β -bromopropionic, or chloromalonic acids. The products are fusible and are soluble in alkali. C. HOLLINS.

[Resinous] condensation product and its manufacture. A. L. BROWN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. Co. (U.S.P. 1,680,408, 14.8.28. Appl., 23.2.22).—Phenol is condensed with formaldehyde and tung oil. C. HOLLINS.

Plastic material from peat, and its manufacture. I. S. MELLANOV, Assr. to KEMIKAL, INC. (U.S.P. 1,681,155, 14.8.28. Appl., 23.2.28).—A solution of peat

in hot aqueous sodium hydroxide is treated with phenol and formaldehyde to give a brown to black plastic mass.

C. HOLLINS.

Preparation of resinous condensation products.

BRIT. THOMSON-HOUSTON Co., LTD., Assees. of [A] L. V. ADAMS and [B] R. H. KIENLE (B.P. 273,748 and 284,349, [A] 1.7.27, [B] 28.1.28. U.S., [A] 1.7.26, [B] 29.1.27).—(A) The heat-hardening of "glyptal" resins is rapidly carried to the completely condensed or C-stage, regardless of the production of cavities. The pumice-like mass is then ground and extracted in a closed container with suitable solvents, *e.g.*, glycol diacetate, ethyl phthalate, acetone oils, toluidine, benzyl alcohol, cresol, indene, etc. (B) Polyhydric alcohols and polybasic aromatic acids are condensed with oxidisable unsaturated fatty acids (*e.g.*, acids derived from drying oils), in the absence of inorganic acids, *e.g.*, sulphuric acid. The products are soluble in drying oils at ordinary temperatures, and can also be fully hardened without baking.

S. S. WOOLF.

Manufacture of artificial resins. I. G. FARBERIND. A.-G., Assees. of M. PAQUIN, A. VOSS, and H. WOHLERS (G.P. 448,427, 5.8.24).—The resins obtained by condensation of acetaldehyde, crotonaldehyde, aldol, etc. are suspended or dissolved in inert media (*e.g.*, alcohol, water) and treated with chlorine or with potassium chlorate and hydrochloric acid. The products are suitable for production of water- and light-fast polishes.

C. HOLLINS.

Preparation of spirit-soluble resins. I. G. FARBERIND. A.-G., Assees. of R. LEOPOLD and A. MICHAEL (G.P. 449,115, 5.5.25).—Transparent yellow to red resins, suitable for spirit varnishes and polishes, are obtained by condensing a vinyl ester (*e.g.*, acetate) with aliphatic aldehydes or substances generating them; *e.g.*, paraformaldehyde at 150° and 20 atm. in absence of oxygen gives a yellow fusible resin. Acetaldehyde, *n*-butaldehyde, paraldehyde, crotonaldehyde, ethylidene diacetate, etc. are also suitable.

C. HOLLINS.

Removing liquid from resinous products. NAUGATUCK CHEM. Co., Assees. of M. G. SHEPARD (B.P. 276,627, 20.6.27. U.S., 25.8.26).—Mechanically retained liquid, formed or used in the preparation of resinous aldehyde-amine condensation products, is removed by melting the latter with a fluxing agent, heating, and agitating until the liquid is eliminated. The liquid-free product is drawn off, cooled, and solidified. As fluxing agents, materials, *e.g.*, stearic acid, compatible with the further use of the resinous material, *e.g.*, in rubber compounding, must be used.

S. S. WOOLF.

Manufacture of shellac-like masses. SIEMENS & HALSKE A.-G. (G.P. 449,275, 29.10.22).—Resins closely resembling shellac in nature and properties are obtained by heating together aliphatic and hydroaromatic hydroxylated acids. For the aleuritic acid of shellac are used lactic, glyceric, dihydroxystearic (from oleic acid), trihydroxystearic (from ricinoleic acid), or tetrahydroxystearic (sativic) acid etc. or the oxidation products from linseed oil acids, and as second component in place of shellolic acid cyclohexanolcarboxylic acids,

tetrahydronaphthoic acids or their hydroxy-derivatives etc. Examples are: lactic acid and methylcyclohexan-4-ol-2-carboxylic acid heated at 120° in a vacuum for 3 hrs. and then with dihydroxystearic acid at 140° for a further 3 hrs.; trihydroxystearic acid heated at 120°, then dissolved in alcohol and saturated with hydrogen chloride, finally heated with trihydroxypalmitic and β -hydroxycamphoric acid at 150° and mixed with beeswax and carnauba wax; sativic acid mixed with hydroxyabietic and lactic acids and melted under reduced pressure with zinc chloride, wax being added after 5–6 hrs.

C. HOLLINS.

Manufacture of urea-formaldehyde condensation products. K. RIPPER, Assr. to F. POLLAK (U.S.P. 1,687,312, 9.10.28. Appl., 20.3.24. Austr., 31.3.23).—See B.P. 213,567; B., 1925, 681.

Removal of scale from boilers (B.P. 281,598).—See XXIII.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Reinforcement of rubber by fillers. J. T. BLAKE (Ind. Eng. Chem., 1928, 20, 1084–1088).—Ingredients which form with the rubber a bond which is stronger than the matrix are classed as reinforcing agents; those giving a bond weaker than the matrix are diluents. Regarding the tensile strength, calculated on the cross-section at break, as a measure of the tensile strength of the matrix, a reinforcing agent such as carbon black does not increase the strength of the matrix; the apparent increase in tensile strength is due to the reduction in elongation. When the proportion of reinforcing agent is increased beyond the stage of reinforcement, perfect dispersion is no longer possible and rupture occurs at the bond between filler and rubber and the elongation is disproportionately reduced. The controlling factor in reinforcement is not particle size, but the surface energy in the filler-rubber bond. An intimate relationship between the natural fatty acids of rubber and the dispersion of a filler such as carbon black is indicated by the fact that the reinforcing effect of carbon black in rubber ceases near the proportion of 30 vols. to 100 of rubber, and that this is in approximate agreement with the maximum proportion of dispersed carbon black over which the natural fatty acids of the rubber could provide a unimolecular film. This result probably explains the improved dispersion of carbon black obtainable by the additional incorporation of stearic acid, pine tar, or reclaimed rubber.

D. F. TWISS.

Dispersion of "pigments" in rubber. I. Microscopical studies of agglomeration and flocculation. E. A. GRENQUIST (Ind. Eng. Chem., 1928, 20, 1073–1078).—The particle size of carbon black in rubber is 40–50 μ . It is difficult to count the particles in the microscope, but from the degree of blackness of mixtures containing 0.5% it is possible to discriminate between various types of amorphous carbon. In a 1% mixture in rubber, zinc oxide shows an average particle size of 0.49 μ . Vulcanisation of mixtures of rubber with sulphur and other ingredients appears to induce an initial improvement in dispersion, followed by flocculation, this being most pronounced with carbon black,

lamp black and thermatomic black exhibiting flocculation to a less degree. In a mixture containing carbon black (2 pts.) and zinc oxide (30 pts.) the latter seems to check the dispersion of the former. Agglomeration of particles of ingredients on the mixing mills usually occurs around any larger nuclei, *e.g.*, of foreign matter, in the rubber. D. F. TWISS.

Light absorption of stretched and unstretched rubber and of isoprene. M. KRÖGER and H. STAUDE (Gummi-Ztg., 1928, 43, 22—24).—In a quartz spectrograph, isoprene and various samples of rubber exhibit only continuous absorption. Synthetic rubber made from isoprene is more transparent than isoprene itself at the same thickness. The transparency of synthetic and also of natural rubber increases with stretching, presumably on account of some molecular change. Rubber vulcanised with sulphur chloride is much less transparent. The relative transparency of stretched rubber to ultra-violet light might lend itself to the production of lenses consisting of spherical membranes of rubber distended with an optically transparent liquid. D. F. TWISS.

Carbon blacks and their use in rubber. III. Ageing effects. L. B. COX and C. R. PARK (Ind. Eng. Chem., 1928, 20, 1088—1091; cf. Goodwin and Park, B., 1928, 579, 649).—Vulcanised mixtures containing various proportions of different types of carbon black have been examined as to the alteration in their physical characteristics after dark storage for 18 months at 18° in moving air. The samples show slight hardening, and the stress-strain curves become straighter. The decrease in tensile strength is distinctly greater for typical carbon blacks than for those, *e.g.*, thermatomic black, made by processes involving thermal decomposition; the ageing of the mixtures vulcanised with the aid of mercaptobenzthiazole or ethyldeneaniline also is superior to those containing hexamethylenetetramine or diphenylguanidine. The relative alteration of the samples with respect to loss in tensile strength and in abrasion resistance and to increase in weight is fairly consistent. The additional presence of stearic acid does not affect the ageing properties of the "compounds," nor does the presence of acidity in the sulphur used, the zinc oxide also present presumably exerting a sufficient neutralising effect. A close connexion appears to exist between the ageing influences of the various blacks and their adsorptive character, possibly because of the different proportions of oxygen thereby introduced into the rubber. D. F. TWISS.

Rubber mixtures containing carbon black. W. ESCH (Gummi-Ztg., 1928, 43, 75—78).—Attention is drawn to the need for care in discriminating between carbon blacks made by the incomplete combustion of natural gas, special blacks made from natural gas by thermal decomposition, and lamp blacks made by the incomplete combustion of oil. There are marked distinctive features; *e.g.*, carbon blacks give rubber mixtures with lower rate of vulcanisation and with higher tensile strength than lamp blacks. D. F. TWISS.

Jelutong. C. D. V. GEORGI (Malayan Agric. J., 1928, 16, 204—211; cf. B., 1928, 309).—Introduction of

soluble iron or copper salts into latex causes a tendency to oxidation in the extracted jelutong; soaking of fresh coagulum in solutions of these salts or of caustic alkali has no such effect. Manganese salts do not induce oxidation, but favour mould development. The type of deterioration suffered by jelutong is influenced by the locality of its origin. D. F. TWISS.

Valuation of jelutong. C. D. V. GEORGI (Malayan Agric. J., 1928, 16, 220—233).—The irregularity of distribution of moisture in a block of jelutong and variation between the blocks themselves render trustworthy evaluation difficult. It is desirable that the weight and proportion of samples to be taken and the actual method of sampling and testing be standardised. D. F. TWISS.

Rubber-like state of matter. VON WEIMARN.—See V.

PATENTS.

Treatment of rubber latex and manufacture of rubber articles from latex. S. M. CADWELL, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,680,856—8, 14.8.28. Appl., [A] 28.8.25, [B, C] 22.9.25. Renewed [B] 19.6.28).—(A) Rubber latex, particularly ammonia-preserved 33% latex which has been twice treated with pectin and creamed, is heated with zinc oxide (3%), sodium acetate (3%), and glue (1%) at 65°. It deposits rubber rapidly on porous articles by the dipping process. In place of sodium acetate a thio-acid salt (*e.g.*, sodium dithiobenzoate) may be used, and the latex vulcanised with sulphur before deposition. (B) A vulcanised latex is obtained by adding sulphur, zinc oxide, and tetramethylthiuram disulphide (sufficient to give a hard rubber below 100°) to a latex which already contains sulphur, zinc oxide, and a non-nitrogenous carbon disulphide accelerator sufficient to give a soft elastic product. (C) A latex treated as in (A) with zinc oxide, sulphur, and sodium dithiobenzoate, with or without glue, is vulcanised at 65°, and rubber articles are made by a dipping process. C. HOLLINS.

Treatment of rubber, and product thereof. H. E. CUDE, Assr. to NAUGATUCK CHEM. CO. (U.S.P. 1,680,862, 14.8.28. Appl., 27.5.27).—Rubber, raw or vulcanised, is incorporated with an organic acid capable of forming a soluble soap (*e.g.*, oleic or stearic acid, sulphonated oils, etc.), and residual acidity is neutralised with alkali. A reclaim rubber is obtained as a plastic mass, or may be emulsified in the usual manner. C. HOLLINS.

Treatment of rubber-fibre waste material. R. P. ROSE, Assr. to MECHANICAL RUBBER CO. (U.S.P. 1,680,915, 14.8.28. Appl., 7.1.28).—The broken-up material is worked with a soap-forming fatty acid to a putty-like consistency, and then with aqueous sodium hydroxide. The rubber is finally recovered as dispersion by beating the product with aqueous alkali and a protective colloid. C. HOLLINS.

Vulcanisation of rubber. R. V. HEUSER, Assr. to A. C. BURRAGE, JUN. (U.S.P. 1,681,806, 21.8.28. Appl., 27.1.21).—Tri-*o*-tolylguanidine is used as accelerator. C. HOLLINS.

Production of coherent caoutchouc or the like products. ANODE RUBBER CO., LTD. From P. KLEIN,

F. GÁBOR, and A. SZEGVÁRI (B.P. 297,127, 16.3.27).—Two or more pieces of material obtained directly as deposition strata from aqueous dispersions, *e.g.*, by electrophoresis, dipping, spreading, or spraying, when brought together in a moist condition, with or without pressure, and then dried, become united and inseparable. The process can be applied to the production of hollow rubber articles from latex and to the attachment of valve patches to air-tubes formed by electrophoretic deposition from aqueous rubber dispersions.

D. F. TWISS.

Vulcanisation of rubber. C. J. T. CRONSHAW and W. J. S. NAUNTON, Assrs. to BRIT. DYESTUFFS CORP., LTD. (U.S.P. 1,687,861, 16.10.28. Appl., 4.1.26. U.K., 12.3.25).—See B.P. 253,197; B., 1926, 682.

Abrasive articles (U.S.P. 1,681,891).—See VIII.

XV.—LEATHER; GLUE.

Action of acids on vegetable-tanned leathers. D. WOODROFFE (J. Soc. Leather Trades' Chem., 1928, 12, 385—389; cf. B., 1927, 757, 790).—A 10% solution of oxalic acid destroyed vegetable-tanned leather soaked therein for 2 months; similarly a 5% solution considerably weakened it. The effect of other acids depended on the p_H value of their solutions, the lower the p_H value, the greater is the weakening effect. The leather was not weakened by immersion in warm solutions of acids (5% or 10%) for $\frac{1}{2}$ hr., washing, and drying out. Vegetable-tanned skivers, East Indian sheep, basils, kip splits, and shoulders, respectively, were treated with *N*-solutions of oxalic acid, ammonia-ammonium chloride, sulphuric acid, and with 1% sulphuric acid for $\frac{1}{2}$ hr., then dried, and kept for 6 months. The thinner the leather the greater was the relative weakening effect of both the acids. The moderate use of sulphuric or oxalic acids, such as might be required in leather dressing, does not damage vegetable-tanned leathers, except skivers.

D. WOODROFFE.

Utilisation of tanning materials containing a large amount of soluble non-tans. V. S. SADIKOV and P. A. YAKIMOV (Trans. State Inst. Appl. Chem. Moscow, 1927, No. 6, 40—63).—The finely-powdered bark of *Saxifraga* (*Bergenia*) *crassifolia* (badan) is treated first with calcium hydroxide, then with cold water, and finally with hot water and oxalic acid or sodium hydrogen sulphate. The first (cold) extract contains 30%, and the second (hot) extract contains 60%, of the tannins. The method has been applied to other Russian tanning materials.

CHEMICAL ABSTRACTS.

PATENTS.

Leather substitute. E. I. DU PONT DE NEMOURS & Co., Assecs. of J. D. MCBURNEY and E. H. NOLLAU (B.P. 273,324, 24.6.27. U.S., 24.6.26).—Textile fabric coated with a cellulose ester, *e.g.*, pyroxylin, is provided with a flexible, waterproof surface comprising casein and glycerol to allow adhesives to spread over and adhere to it.

L. A. COLES.

Manufacture of artificial [horn-like] masses containing casein. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,483, 23.5.27).—Solid or

liquid hydroaromatic or aliphatic-hydroaromatic amines or aliphatic amines containing hydroxyl groups, or their salts or derivatives, are incorporated with casein.

L. A. COLES.

Mineral tanning and products obtained thereby. F. W. WEBER (B.P. 297,754, 21.3.27).—See U.S.P. 1,642,054; B., 1927, 917.

XVI.—AGRICULTURE.

Soils. III. Absorbing power of soils for ammonia. J. CLARENS and (Mme.) PÉRON (Bull. Soc. chim., 1928, [iv], 43, 962—969; cf. B., 1927, 950).—The absorption of ammonium salts by a clay soil containing considerable quantities of lime and a small amount of humus is investigated. True adsorption does not occur, since the absorption takes place in stages. The order of absorbability is hydroxide > phosphate > sulphate > chloride. The bearing of these results on the artificial manuring of the soils is discussed. O. J. WALKER.

Losses and gains of nitrogen in an Indian soil. H. E. ANNETT, A. R. P. AIYER, and R. N. KAYASTH (Mem. Dept. Agric. India, 1928, 9, 155—232).—Six plots on the black cotton soil of Nagpur were sampled at weekly intervals for two years. Determinations of nitrate on these samples were made by the phenol-disulphonic acid method. A rapid accumulation of nitrates took place during the June and July rains. Heavy rains in August washed these nitrates through the soil, and the nitrate content of the soil then remained at a low level till January. The cultivation of cold-weather crops was then accompanied by a further rise in the nitrate content of the soil. Although these losses of nitrates were accompanied by rises in the nitrate content of drainage and well waters, it is concluded that some denitrification must take place in order to account for the large amounts of nitrates lost. In spite of this heavy loss of nitrogen, the total nitrogen content of the soil now is the same as it was 25 years ago. Considerable fixation of nitrogen must therefore have taken place. II. J. G. HINES.

Forms and properties of water-soluble phosphorus in soils. F. W. PARKER (Science, 1928, 67, 402—403).—The presence of at least two forms of organic phosphate in soil extracts and solutions is indicated; the amounts of the two forms are usually approximately equal. Soil extracts and solutions can be boiled for 2 hrs. under reflux without materially increasing the inorganic phosphate content.

A. A. ELDRIDGE.

Electrodialysis of soil. K. N. TARANOV (Ukrainian Chem. J., 1928, 4, [Tech.], 89—112).—Various Russian soils were subjected to electrodialysis, which is shown to be a more efficient means of separating inorganic anions and cations than extraction with ammonium chloride solution. The time necessary to complete the process varied from 19 hrs. for one soil to 152 hrs. for Ukrainian black earth. The acidity of the soil rose considerably after electrodialysis, in one case from 15 to 174 c.c. of 0.1*N*-acid. This increase in acidity applies both to total acidity as measured by extraction with *N*-sodium acetate, and to replaceable acidity as measured by titration of the potassium chloride extract with 0.1*N*-

alkali. This suggests the presence of two different types of substance in the soil, the relative ratio of which determines its eventual acidity. The soil residue after electrodialysis was practically non-hygroscopic, pointing to an aggregation of colloidal particles under the influence of this process.
R. TRUSZKOWSKI.

Determination of the electrical conductivity of the aqueous extract of soil as a rapid means of detecting its probable fertility. A. SEN (Mem. Dept. Agric. India, 1928, 9, 247—254).—The sieved air-dry sample is placed in a tube, 5 pts. of conductivity water are added, and the tube is closed by a paraffined cork carrying a dip electrode. The tube is then shaken and placed in a thermostat at 30°. After 2 hrs. the conductivity is measured. Similar measurements are made at intervals up to 9 days. Fertile soils show a rapid increase in the conductivity of the extract; infertile soils show little or no change. The comparison is, of course, limited to soils from adjacent areas.

H. J. G. HINES.

Law of crop yield. A. RIPPEL (Z. Pflanz. Düng., 1928, 12A, 38—55).—Oats, mustard, and sunflowers were grown simultaneously in pots and fertilised with increasing amounts of nitrogen and potash. On applying the Mitscherlich formula to the crop yields it was shown that the nutrient constant varied from crop to crop. It is concluded that the factors influencing crop yield cannot be combined in a single simple expression of the Mitscherlich type.

H. J. G. HINES.

The hot-fermentation process [of manure] and "edelmist." G. RUSCHMANN (Z. Spiritusind., 1928, 51, 258—259, 266—267, 275—276, 284).—The Krantz process for the hot fermentation of manure is described. The humification process in hot-fermented stacks is more complete than in ordinary yard stacks, there being practically no fibrous matter remaining. Comparison of the carbon dioxide production in soil composts with manure stacked and fermented in different ways indicates that the ageing of "edelmist" does not result in appreciable losses of easily oxidisable carbonaceous matter. The importance of close adherence to the Krantz procedure is emphasised. Unless the initial fermentation temperature of 60—70° is reached, the second stage or ripening of the stack involves loss of both organic matter and nutrient material.

A. G. POLLARD.

Artificial farmyard manure. W. R. GREENSTREET (Malayan Agric. J., 1928, 16, 194—203).—Under the conditions obtaining in Malay, vegetable matter will rot easily and quickly provided that acidity is neutralised, a free supply of air is given, and the moisture content maintained at 75%. Although the claims made for the "Adco" process are substantiated, the addition of "Adco" reagents to the materials tested would appear to be superfluous.

H. J. G. HINES.

Basic slag. WEISS.—See X. **Fruit of *Schinus molle*.** CREMONINI.—See XX.

PATENTS.

Mixed fertilisers. J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 297,960, 15.9.27).—The fertilisers comprise potassium nitrate and diammonium phosphate.

L. A. COLES.

Dry fungicide. E. MOLZ, Assrs. to CHEM. FABR. L. MEYER (U.S.P. 1,685,715, 25.9.28. Appl., 8.9.25. Ger., 24.9.24).—A mixture of mercuric chloride and alkali iodide.

F. G. CROSSE.

Insecticide and fungicide. F. M. ROGERS and C. P. McNEIL, Assrs. to STANDARD OIL Co. (U.S.P. 1,679,919, 7.8.28. Appl., 3.5.26).—Paraffin oil, an alkali salt of sulphonated mineral oil ("mahogany soap"), and a copper naphthenate or mineral-oil sulphonate are emulsified in water with addition of a little alcohol, oleic acid, and alkali if desired.

C. HOLLINS.

Protection of plants from animal pests. CHEM. FABR. DR. H. STOLTZENBERG, Assees. of R. JANISCH (G.P. 448,446, 18.4.25).—Furfuraldehyde, solubilised with glue, is sprayed in the form of aqueous solution containing also contact or respiratory poisons.

C. HOLLINS.

Amines of hydroaromatic-aliphatic series (B.P. 297,484).—See III.

XVII.—SUGARS; STARCHES; GUMS.

[Liming of diffusion juice.] H. CLAASSEN (Z. Ver. deut. Zucker-Ind., 1928, 543—548).—The analytical results from a number of molasses are calculated to a basis of non-sugar constituents. The results indicate a lower lime content and colour when dry-liming is practised.

F. E. DAY.

XVIII.—FERMENTATION INDUSTRIES.

Kilning of malt. R. SCHLENK (Woch. Brau., 1928, 45, 415—418, 426—430, 436—440, 451—456).—A mathematical treatment of kilning conditions, based on Mollier's diagrammatic representation of the properties of air at varying temperatures and moisture content. From the data given the relations between rate of drying and air and malt temperatures and moisture contents are deduced. Since the evaporation cools the malt its temperature is much below that of the air in the earlier stages. As the moisture in the malt falls below 25% the hygroscopic effect becomes increasingly important. Neither this effect nor that due to varying surface is constant for different malts.

F. E. DAY.

Nitrogenous constituents of wort assimilable by yeast. W. WINDISCH, P. KOLBACH, and E. HENNECKE (Woch. Brau., 1928, 45, 389—393, 399—406, 409—415, 421—426, 431—435).—If wort is diluted till it contains about 2.7% of solids, inoculated with a trace of yeast, and after 48 hrs. at room temperature incubated for 7 hrs. at 30° with aeration, almost all the assimilable nitrogen is removed. When the amino-nitrogen in worts before and after fermentation is determined by the Van Slyke and Sørensen's formol titration methods, the latter gives higher values in both cases. The decrease in amino-nitrogen during fermentation as determined by these methods accounts for about 60% of the nitrogen assimilated by the yeast. Since by adsorption on asbestos-kaolin or by ultrafiltration, as much as 25% of the total but no assimilable nitrogen is removed, the more complex proteins are not assimilable, neither are peptones, since the action of papain or pepsin does not increase the amount of nitrogen assimilated. The action of trypsin, the proteolytic enzyme of malt, and of acid

hydrolysis increases proportionally the amounts of both assimilable and amino-nitrogen, and the maximum action of each agent is shown by both after equal times of digestion. Formol titration in stages indicates that the ratio of polypeptides to amino-acids is about the same before and after fermentation, hence both are assimilable.

F. E. DAY.

Influence of various yeast-races and temperatures on fermentation and quality of beer.

R. HORCH and SCHULTEIS (Woch. Brau., 1928, 45, 394—398).—Similar worts were fermented with three different bottom-fermentation yeasts, two of which flocculated normally, and the other settled in a powdery form ("Staubhefe"). The fermentations were repeated, employing pitching temperatures of 3.5°, 4.5°, and 5.6°, and maximum temperatures of 6.5°, 7.5°, and 9° respectively. The temperature, saccharometer readings, and p_H values are recorded for each fermentation, and from these and the character of the resulting beers it is concluded that lower temperatures are favourable to the quality of the beer, the best pitching temperature being about 4°, the maximum during fermentation 7—8°. Since a higher degree of fermentation is given by the "Staubhefe" than by the others, it is suggested that the condition and stability of beers might be improved by fermenting as usual with a flocculating yeast and later adding a yeast of higher fermenting power to induce a vigorous secondary fermentation.

F. E. DAY.

Comparative determinations of hydrogen-ion concentration of worts and beers by the hydrogen and quinhydrone electrodes.

F. STOCKHAUSEN and E. F. ROTHENBACH (Woch. Brau., 1928, 45, 459—462).—The hydrogen electrodes employed were those of Michaelis and of Lüers and gave results in good agreement on a series of worts and beers. The quinhydrone electrodes of Trénel and of Lüers gave results which differed from them in some cases by as much as p_H 0.23, generally towards the acid side. It is concluded that though the quinhydrone electrode is sufficiently accurate for brewery routine purposes, if due regard is paid to the fact that its readings in different liquids are not strictly comparable, it should not be used with worts or beers if scientifically accurate results are required.

F. E. DAY.

Formol titration [of beers etc.]. P. KOLBACH (Woch. Brau., 1928, 45, 446—451).—In carrying out the formol titration on beers and worts, the phosphates are first removed by precipitation with barium chloride in alkaline solution. The p_H of the filtered liquid is first adjusted to neutrality against a phosphate buffer by employing neutral-red as indicator in a Lüers acidimeter. After addition of formaldehyde, it is titrated to p_H 9, against a borate buffer, using phenolphthalein as indicator. Precautions are taken to maintain correct concentrations of wort, indicators, and water at the comparison points.

F. E. DAY.

Volatility in steam of certain œnologic acids.

P. JAULMES (Ann. Falsif., 1928, 21, 384—390).—After passing 100 c.c. of water as steam through 10 c.c. of wine, 87% of the benzoic acid and 50% of the salicylic acid present were carried over, the volume of the wine being kept constant. If the wine was benzoated to the

extent of 1% the correction was 0.26 g. (when sulphuric acid had been added and the technique of Blarez followed), and for the addition of 0.5% of salicylic acid the correction was 0.09. The effect of lactic acid produced during "malo-lactic retrogradation" is insignificant. If distillation by Duclaux's method is used, the correction for benzoic acid is 0.151 and for salicylic acid 0.037. The conclusion of Fonzes-Diacon (B., 1928, 686) that salicylic acid should be used as stabiliser for wines is confirmed.

D. G. HEWER.

Butanol [butyl alcohol] fermentation process.

G. L. GABRIEL (Ind. Eng. Chem., 1928, 20, 1063—1067).—A general description of the factory of the Commercial Solvents Corporation of Maryland is given, together with a survey of the development of the butyl alcohol industry as an offshoot from the original acetone production.

A. G. POLLARD.

Hydrogen sulphide in fermentation carbon dioxide.

WANDERSCHECK (Woch. Brau., 1928, 45, 441—446, 463—468).—The hydrogen sulphide liberated by yeast during fermentation is determined by absorption in lead nitrate solution, dissolving the precipitate in aqua regia, and colorimetric determination of the lead as sulphide. A series of worts, containing about 11% of solids, fermented at 25° for 5 days, gave the following results. The amount of hydrogen sulphide varies very widely, even with worts from similar malts; thus unhopped Pilsener worts yielded 7.03—16.52 c.c. and hopped worts 8.37—16.26 c.c. per hectolitre. Highly attenuative yeasts yield more than yeasts of low attenuative power, but the proportions from different worts are constant. No appreciable amount of hydrogen sulphide is produced during yeast autolysis. Though the malt is the chief source of the hydrogen sulphide, none is evolved during germination. Part of the sulphur compounds of malt are precipitated during boiling, hence addition of the deposit produced to a subsequent fermentation raises the amount of hydrogen sulphide, whilst the addition of hops to the boil reduces it. This latter effect is masked if sulphured hops are used. The addition of sodium, magnesium, and calcium sulphates lowers, leaves unchanged, and raises the amounts of sulphide, respectively. The amount is also increased by the addition of free sulphur or of sulphurous acid, also by high temperature and acidity; low temperature and alkalinity have the reverse effect. The amount of yeast added is without influence, but yeasts cultivated at any temperature do not reach their maximum hydrogen sulphide production at another temperature until after several cultivations. Experiments in the brewery give results comparable with those obtained in the laboratory with the same worts, and indicate that (in bottom-fermentation) hydrogen sulphide begins to be evolved during the third day, and that the amount increases up to about the sixth day, or frothing stage, after which it decreases rapidly, but is continued slowly during the after-fermentation. The bearing of the results on brewing practice is fully discussed.

F. E. DAY.

The Gutzeit test [for arsenic in brewing materials]. A. D. COMRIE and T. J. WARD (J. Inst. Brew., 1928, 34, 530—533).—The test may be applied to the

examination of brewing materials with equal accuracy to the Marsh-Berzelius method, and without susceptibility to many disturbing factors possessed by the latter. Since the organic matter present in malt is more or less constant in composition, its destruction prior to the determination of arsenic by the Gutzeit method may be omitted. The arsenic is determined in the untreated malt and the content so obtained corrected by multiplying by the factor 1.3. On the other hand beer, hops, grains, etc., owing to the retarding effect of their constituents on the evolution of the gas, as a preliminary to the Gutzeit test, should be evaporated to dryness with a 20% solution of magnesium nitrate, and the residue ignited until free from carbon. C. RANKEN.

Hot fermentation of manure. RUSCHMANN.—See XVI.

PATENTS.

Isolation of nucleic acids from yeast. EINSIEDLER BRAUHAUS, Assecs. of H. HAEHN (G.P. 448,947, 12.10.24).—Pressed yeast is stirred with hot concentrated sodium hydroxide solution, and glacial acetic acid is added until faintly acid (p_H 5—6). The yeast is removed by centrifuging and nucleic acids are precipitated as ferric salts. These are treated with 10% sodium acetate, ferric hydroxide is removed by filtration, and nucleic acids are precipitated from the filtrate by addition of alcohol and acid. C. HOLLINS.

Production of lipase. S. YAMAMOTO (U.S.P. 1,687,050, 9.10.28. Appl., 9.6.25. Japan, 16.8.24).—See B.P. 238,507; B., 1925, 890.

Tartaric acid and salts (F.P. 624,540).—See VII.

XIX.—FOODS.

Composition of sheep's milk. R. MARTIN (Ann. Falsif., 1928, 21, 390—397).—Analytical figures for a series of morning and evening milks from individual Larzac sheep from Roquefort show that the C.M.S. ("simplified molecular constant") is higher (a mean of 80.5 for 97 milks) than that of cows' milk (with a mean of 73—74), owing particularly to the greater proportion of chlorides present, and the freezing point is correspondingly lower (for cows' milk between -0.55° and -0.56° and for sheep's milk between -0.58° and -0.611°). The original acidity of sheep's milk is higher than that of cows' milk, since the proportion of casein is higher. With certain exceptions, the serum of the milk and the blood of a female in milk are distinctly isotonic, and differences of the same order are present in the blood sera of sheep and cattle as in the milk sera. D. G. HEWER.

Use of starch for characterisation of margarine. E. VIROUX (Ann. Falsif., 1928, 21, 420—424).—The starch used (e.g., potato or rice starch) is best incorporated in the margarine in the mixture of fats. In order to determine the amount of starch present, light petroleum is added to the sample of margarine, and the water layer is separated, deprived of all fat, and the invert sugar present determined. The aqueous extract from another sample is made up to known volume, acidified with sulphuric acid, boiled under reflux, neutralised, 60% methyl alcohol is added, and the mixture is cleared, made up to volume, and titrated with Fehling's solution.

The content of invert sugar is deducted from the total, and the difference multiplied by 0.9 gives the amount of starch added. In order to detect starch in butter to which margarine containing 0.2% of starch has been added, the water from a 50 g. sample is separated by centrifuging, deprived of all fat, and, after addition of 60% methyl alcohol, centrifuged for 15 min. The casein holding the starch is thus agglomerated, and, after pouring off the clear liquid, 5 c.c. of 95% alcohol are added to dehydrate the casein and allow it to be completely extracted by ether. It is then suspended in warm water, dissolved by addition of 1 c.c. of ammonia, and, after centrifuging, the starch from the original 50 g. is left at the bottom of the tube mixed with a little casein, which is removed by further washings with ammonia solution. The starch is examined microscopically and the addition of 1% of margarine to butter may thus be detected. D. G. HEWER.

The formol tests for distinguishing artificial from natural foodstuffs. H. E. HILL (Chem. Eng. and Min. Rev., 1928, 20, 401—402).—An adaptation of Sörenson's formol titration for proteins in which solutions of foodstuffs and formaldehyde, both carefully neutralised with sodium hydroxide to a defined pink coloration of phenolphthalein, are mixed, the degree of resultant acidity being determined (cf. B., 1927, 456), was used in respect to fruit juices in beverages. Juices of various fruits gave titrations which are of the same order in the different species, and the method appears to be capable of considerable applications. When applied to vinegars the results correlated closely with the nitrogen and phosphorus contents of the samples. C. A. KING.

Butter fats. ATKINSON. **Butter fat in margarine.** KNETEMANN.—See XII.

PATENTS.

Neutralisation of acidity or alkalinity of decomposition products [foods] by glutamic acid or its salts. M. FUJII (U.S.P. 1,680,865, 14.8.28. Appl., 3.8.26. Japan, 7.8.25).—To food products prepared by alkaline treatment of proteins, carbohydrates, etc., glutamic acid, its hydrochloride, or calcium glutamate is added to neutralise free alkali, the sodium glutamate formed being pleasant-tasting and nutritious.

C. HOLLINS.

Manufacture of cattle foodstuffs. W. ELLIS (B.P. 297,235, 26.10.27).—A mixture of substances insoluble in water or animal saliva, e.g., calcium phosphate, with others which are soluble, e.g., sodium chloride, sugar, or glucose, is compressed into a form suitable for consumption by chewing or licking. F. R. ENNOS.

Preparation of a foodstuff for feeding animals. A. EHRENREICH (B.P. 284,339, 27.1.28. Belg., 28.1.27).—The body or cuttings of fish such as *Chondropterygii selachii* or more particularly the plagiostomi such as the shark are disintegrated, and the pulp is desiccated *in vacuo* at $50-60^\circ$. F. R. ENNOS.

Manufacture of feeding cakes. C. N. KJAERGAARD (B.P. 297,596, 23.11.27).—The organic waste materials, which may contain adhesive substances, are dried at 55° to a water content of not more than 16% and then compressed at 350 atm. H. ROYAL-DAWSON.

Conservation of food etc. (B.P. 297,074).—See III.
Butter from buttermilk (U.S.P. 1,683,728—9).—See XII.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Physical determinations of some iodine preparations used in pharmacy. E. CANALS and P. SUIFFET (J. Pharm. Chim., 1928, [viii], 8, 308—313).—The Tyndall effect, Brownian movement, ionisation factor, electric charge, and behaviour on dialysis and ultrafiltration of the following preparations, each containing 0.005% I, have been examined: iodine in aqueous potassium iodide, a mixture of tincture of iodine and water, iodised water without potassium iodide, iodised tannin, iodine gum, iodine jelly, iodised gelatin, "sol lumière," and "sol gautrelet." With the possible exceptions of iodised water and the tincture of iodine and water mixture, the iodine was not present in the colloidal state.
E. H. SHARPLES.

Rapid method for differentiating tartaric and citric acids. L. ROSSI (Annali Chim. Appl., 1928, 18, 366—368).—A drop of the freshly prepared aqueous solution of the acid to be tested is added to 0.1—0.2 g. of solid ammonium metavanadate. On stirring, an orange-red polyvanadate is formed in the case of tartaric acid, whilst with citric acid the liquid becomes yellow. If excess of either tartaric or citric acid solution is heated with 1 c.c. of cold saturated vanadic acid solution, the blue colour of the vanadyl cation appears; this reaction proceeds also in the cold, but more slowly.
T. H. POPE.

Antipyrinum coffeinocitricum. G. MIKÓ (Ber. ungar. pharm. Ges., 1926, 2, 88—96, 216—217; Chem. Zentr., 1928, i, 1563).—For the identification of the caffeine by the murexide test, less than 25% of antipyrine must be present. The separation is effected by shaking with 96% alcohol or, better, with amyl alcohol; the residue is washed with ether and oxidised with 3% hydrogen peroxide solution and a few drops of 5*N*-hydrochloric acid.
A. A. ELDRIDGE.

Evaluation of theobrominum natriosalicylicum. G. MIKÓ (Ber. ungar. pharm. Ges., 1927, 3, 127—129).—An aqueous solution of the material is treated with a mixture of alcohol (96%) or alcohol and ether with hydrochloric acid, the precipitated theobromine being washed and weighed. For the residue the murexide test is employed.
A. A. ELDRIDGE.

Evaluation of ergot of rye. P. LIPTÁK (Ber. ungar. pharm. Ges., 1926, 2, 211—216; 1927, 3, 426—430; Chem. Zentr., 1928, I, 1562—1563).—Determinations were made of the acid value, activity, and alkaloid content. If the acid value is greater than 2 the activity is subnormal.
A. A. ELDRIDGE.

Evaluation of ergot of rye. B. GAAL (Ber. ungar. pharm. Ges., 1928, 4, 32—42; Chem. Zentr., 1928, I, 1563).—A precipitate, the origin of which was not discovered, was obtained when the ethereal acid extract of physiologically inactive samples was rendered alkaline.
A. A. ELDRIDGE.

Evaluation of guarana. G. and I. MIKÓ (Ber. ungar. pharm. Ges., 1926, 2, 8—13; Chem. Zentr., 1928,

I, 1560—1561).—The caffeine content is determined by finding the smallest quantity of substance which, after extraction with ammonia and chloroform and oxidation of the residue (obtained by evaporation of the filtered solution) with hydrochloric acid and hydrogen peroxide, yields an orange-yellow coloration.
A. A. ELDRIDGE.

Fruit of *Schinus molle*. A. CREMONINI (Annali Chim. Appl., 1928, 18, 361—365).—As was stated by Spica and Arata (Gazzetta, 1884, 14, 199), the ripe fruit of *Schinus molle* contains no piperine, although various text-books still contain statements to the contrary. The fruit contains also a small proportion of a very bitter compound of unknown composition, and a glucoside, to be investigated further. The ethereal oil contains β -phellandrene, pinene, carvacrol, and possibly limonene and dipentene, but not thymol. The presence of the ferrous ion in considerable amount indicates that the fruit may contain aero-oxydase.
T. H. POPE.

Medicinal charcoal. SABALITSCHKA and OEHLKE.
Sedimentation analysis. VON HAHN and THÖLCKE.—See I.

PATENTS.

Manufacture of solid alcoholic solutions of free iodine. K. JUNGMAHN and O. KOLBERT (B.P. 277,953, 7.9.27. Ger., 21.9.26).—"Solid" alcoholic iodine solutions consisting, e.g., of a solution of iodine in alcohol solidified with sodium stearate or palmitate, are stabilised by the presence of iodides (e.g., sodium iodide).

B. FULLMAN.

Production of inactive menthol (the racemate of natural menthol). RHEINISCHE KAMPFER-FABR. G.M.B.H. (B.P. 285,403, 4.4.27. Ger., 15.2.27; cf. B.P. 285,394; B., 1928, 691).—Inactive *neo*- and *neois*-menthols, obtained by the process of the prior patent, are isomerised by the action of hydrogen and nickel or by heating the sodium (etc.) salts, or are oxidised to inactive menthone or *isomenthone* which is reduced with hydrogen and nickel. *dl*-Menthol, m.p. 34—36°, is isolated from the products by the methods of B.P. 289,125 (B., 1928, 547).
C. HOLLINS.

Preparation of aromatic [hydr]oxyaldehydes [vanillin]. J. D. RIEDEL A.-G. (B.P. 285,451, 20.12.27. Ger., 17.2.27).—Alkali salts of *isoeugenol* are oxidised by nitrobenzene at 150° to vanillin, which, after removal of the aniline formed, is separated as bisulphite compound and distilled. 4-Hydroxy-3-ethoxypropenylbenzene, *isochavibetol*, 4-hydroxy-3-(methoxymethoxy)propenylbenzene, and other mono-ethers of 3:4-dihydroxypropenylbenzene may be similarly oxidised.
C. HOLLINS.

Manufacture of urethanes [of nuclear-substituted benzyl alcohols]. I. G. FARBERIND A.-G. (G.P. 448,694, 25.3.25).—*o*- and *p*-Methylbenzyl alcohols are converted by carbonyl chloride in benzene at 0° in presence of dimethylaniline into the corresponding *chloroformates*, from which by the action of ice and ammonia *o*-methylbenzylurethane, m.p. 86—87°, and *p*-methylbenzylurethane, m.p. 122—123°, are obtained. These are almost tasteless and have much stronger antispasmodic action than benzylurethane, and have none of the toxic effects of side-chain-substituted benzylurethanes.
C. HOLLINS.

Manufacture of terpin [hydrate] from nopinene. G. AUSTERWEIL (Swiss P. 117,777, 18.12.25).—Nopinene (β -pinene) is treated with an equal weight of 45% sulphuric acid; preferably concentrated acid is added to nopinene and water. After 5–6 hrs. the mixture is poured into ice-water, and terpin hydrate is removed after keeping 10 hrs. C. HOLLINS.

Manufacture of camphene and derivatives. L. PEUFAILLIT and G. AUSTERWEIL (F.P. 563,208, 20.5.22).—Pinene or turpentine is heated at ordinary or increased pressure with weak acids, such as abietic acid, colophony, or other resin acids, in presence or absence of an inert solvent (benzene or xylene). An unstable compound is formed which readily splits into camphene and abietic acid. The camphene may be frozen out or converted into bornyl and isobornyl acetates. C. HOLLINS.

Manufacture of chloriodo-compounds of the quinoline series. CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING), Assees. of M. DOHRN and A. THIELE (G.P. 447,538, 2.7.24).—Quinolinecarboxylic acids or esters are stirred with iodine mono- or tri-chloride in acetic acid at 50–70°. Atophan forms a *chloriodide*, m.p. 237–238°; *chloriodides* are also obtained from amyl 2-phenylquinoline-4-carboxylate and quinaldine-4-carboxylic acid, m.p. 133–135° and 147–148°, respectively. The compounds lose both halogens when boiled with water. C. HOLLINS.

Manufacture of *N*-acyl derivatives of 3-amino-4-hydroxybenzene-1-arsenoxide and dichloroarsines. POULENC FRÈRES, E. FOURNEAU, and J. TREFOUEL (F.P. 606,238, 19.2.25).—*N*-Acyl derivatives of 3-amino-4-hydroxybenzenearsinic acid are converted by sulphur dioxide and iodine in presence of concentrated hydrochloric acid into the corresponding phenyl-dichloroarsines, and these by treatment with ammonium carbonate give the arsenoxides. Formyl, carbethoxy-, and acetyl derivatives are described. C. HOLLINS.

Manufacture of an organic bismuth-arsenic compound. C. LEVADITI (F.P. 609,146, 31.10.24).—A 15% aqueous solution of sodium 3-acetamido-4-hydroxybenzenearsinate is added gradually at 80–90° to a solution of sodium or potassium hydrogen tartrate containing 30% Bi. Bismuth 3-acetamido-4-hydroxybenzenearsinate is precipitated. C. HOLLINS.

Extracting the cardio-active substance of *Bulbus scillæ*. CHEM. FABR. VORM. SANDOZ (B.P. 287,147, 16.3.28. Ger., 16.3.27).—Fresh squill or its juice is mixed with a readily soluble salt (ammonium or magnesium sulphate) and extracted with an organic solvent immiscible with water (methyl or ethyl acetate) which may contain an aromatic carboxylic acid and/or a small amount of a water-miscible solvent. Dried squill is treated with water and then as above. The active glucoside is recovered by almost complete evaporation of the solution and precipitation with ether. Part of the product is crystallisable, and the rest is freed from tannin-like impurities by treatment with tannin-precipitating agents. B. FULLMAN.

Manufacture of a difficultly water-soluble glucoside of *Adonis vernalis*. F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 288,129, 5.1.28. Ger., 1.4.27. Addn. to B.P. 265,941; B., 1927, 797).—The dark-brown oil obtained in the process of the prior patent is treated with water and with ether to remove the easily soluble glucoside etc. The residue is extracted with chloroform, and the chloroform solution shaken out with water, concentrated, and poured with stirring into light petroleum. A difficultly water-soluble glucoside is precipitated, which is purified by dissolution in 50% alcohol and precipitation of impurities from the solution with lead acetate. The solution is treated with sodium phosphate to remove excess of lead, and the alcohol removed *in vacuo*. The residual oil is dissolved in chloroform and the solution added to light petroleum, when the glucoside is obtained as a yellowish powder. B. FULLMAN.

Treatment of animal glands etc. to increase the activity of the hormones. G. EICHELBAUM and J. ALTENBURG (B.P. 275,183, 21.6.27. Ger., 28.7.26).—Glands and other animal organs having internal secretions are treated (preferably after comminution or reduction to a dry powder and possibly admixture with, e.g., kieselsguhr) with ultra-violet light, with movement or stirring, and with or without the use of a heat filter. The substances and their extracts acquire increased efficiency. [Stat. ref.] B. FULLMAN.

Preparation of vaccines from bacterial poisons. I. G. FARBENIND. A.-G., Assees. of R. BIELING and K. JOSEPH (G.P. 447,813, 16.9.24).—The total bacterial poisons, precipitated from the culture in known manner, are subjected to a 30% formaldehyde treatment or a heat treatment or both. This has the advantage of eliminating other poisons, such as toxones, from the specific toxin, and enables a much more rapid preparation of the vaccine to be made. Thus a beef broth containing 1% of peptone is inoculated with diphtheria bacilli. After 7 days 1 c.c. of a 1% dilution of the culture kills a 250 g. guinea-pig in 4 days. The alcohol precipitate of the culture has the same activity at 0.01% dilution. The precipitate in 0.1% solution is treated at 37° with 0.3% of 30% formaldehyde. 1 c.c. of the liquid after 3 days is easily tolerated by guinea-pigs. C. HOLLINS.

Isolation of alcohols and phenols from mixtures. A. DEPPE SÖHNE, and O. ZEITSCHEL (G.P. 448,419, 14.12.24. Addn. to G.P. 444,640. Cf. B.P. 252,570; B., 1926, 691).—Further to the B.P., crude Japanese peppermint oil containing 44.5% of free alcohols (as menthol), warmed with boric anhydride at 80–100° under reduced pressure, gives an alcohol, m.p. 15°, d_{20}^{25} 0.903, n_D^{20} 35.8° in 1 dm. tube. Thyme oil containing 25% of phenols gives a mixture of thymol and carvacrol with 30% of borneol and linalool. C. HOLLINS.

Manufacture of cosmetic preparations and the like. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 249,130, 16.3.27; cf. B.P. 292,655; B., 1928, 681).—Cosmetic preparations comprising the customary constituents are improved by the inclusion of sorbitol

or its dehydration products or of ether or ester derivatives of either. D. F. TWISS.

Mono[hydr]oxyphenylalkyl ketone derivatives. H. LEGERLOTZ (B.P. 297,756, 22.4.27).—See U.S.P. 1,680,055; B., 1928, 857.

Substituted 4 : 4'-dihydroxybisacylaminoarsenobenzenes [3 : 3'-diacylamino-4 : 4'-dihydroxyarsenobenzenes]. L. BENDA, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,680,845, 14.8.28. Appl., 23.5.27. Ger., 28.6.26).—See B.P. 296,327; B., 1928, 857.

Unsaturated hydrocarbons (F.P. 583,341). **Pharmaceutical preparations** (B.P. 297,074). **Naphthastyril** (B.P. 281,257).—See III. **Nucleic acids from yeast** (G.P. 448,947).—See XVIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Sensitising ripening nuclei of gelatin. A. STEIGMANN (Kolloid-Z., 1928, 46, 57—59).—Observations are described which show that Sheppard's silver sulphide nucleus theory of photographic sensitisation is not sufficiently comprehensive. E. S. HEDGES.

Tartrates and borotartrates as [photographic] restrainers. L. LOBEL (Bull. Soc. franc. Phot., 1928, 15, 167—169).—The addition of tartaric acid and borotartaric acid, prepared by admixture of equimolecular quantities of tartaric and boric acids, to a metol-quinol developer caused retrogression of inertia and apparent decrease in sensitivity resembling the effect of addition of alkali bromide. J. W. GLASSETT.

Measurement of rates of detonation of explosives. JONES.—See XXII.

PATENTS.

Desensitising photographic plates or films. I. G. FARBENIND. A.-G. (B.P. 292,140, 23.5.28. Ger., 15.6.27).—*iso*Phenosafranines, *e.g.*, 1 : 3-diaminophenylphenazonium chloride or 2 : 3-diaminophenylphenazonium nitrate, at 0.02% concentration, are used for the desensitising of plates and films before development. The dye can be washed out of the gelatin and does not stain the hands. C. HOLLINS.

XXII.—EXPLOSIVES; MATCHES.

Photographic study of detonation in solid explosives. I. Development of a photographic method for measuring rates of detonation. E. JONES (Proc. Roy. Soc., 1928, A, 120, 603—620).—The explosive is fired behind a narrow slit in a steel screen, so that the cartridge is obscured with the exception of a narrow central strip of which the width is adjusted so as to give an image about 0.007 cm. thick. In this way the width of the image can be made as small as desired without limiting any dimension of the cartridge, and, as the explosive can be fired much closer to the lens and camera than in previous methods, there is a corresponding increase in the intensity of an image of similar magnifica-

tion. In a cylindrical cartridge of uniform composition, detonating axially, the detonation front is symmetrical about the axis of the cartridge, so that the progress of detonation along any narrow longitudinal strip of the cartridge correctly reproduces that along the whole surface. The effect of the surrounding medium is considered in detail. It is found that, under certain conditions and in certain directions, the disturbance outside a cartridge of solid explosive detonated in a gaseous medium can have a considerably higher speed than that of the detonation zone in the cartridge itself, and in this case the photographic record of the detonation phenomenon may be distorted. A method for overcoming this effect is described. The contour of the disturbed region surrounding a particular cartridge of explosive has been approximately determined at definite intervals from the moment of initiation of the disturbance. The values for the velocity of detonation obtained by the photographic method are in substantial agreement with those from the Dautriche method. Results are tabulated to illustrate the two velocities at which detonation is capable of propagation in the gelatinous explosives and the change in velocity on storing; also the effect of the nitroglycerin content of a powder explosive on its detonation velocity. A method is given for determining the approximate duration of the detonation phenomenon over a transverse section of a cartridge. The stable form assumed by the detonation front inside a cartridge (7.6 cm. diam.) of a modified Glasgow dynamite has been determined. L. L. BIRCUMSHAW.

PATENTS.

Finely-divided nitroguanidine and its manufacture. A. P. TANBERG and R. L. KRAMER, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,679,752, 7.8.28. Appl., 6.4.22).—Nitroguanidine, free from sulphuric acid and in a form suitable for propellant powders, is obtained by saturating water at 140—160° with recrystallised nitroguanidine and discharging the solution into ice and water. Water dissolves 9.09% of nitroguanidine at 100°, 60% at 146°, 96.5% at 158°.

C. HOLLINS.

Explosives. IMPERIAL CHEMICAL INDUSTRIES, LTD., and T. J. R. ALEXANDER (B.P. 297,375, 20.6.27).—A mixture of ammonium perchlorate and sodium nitrate is prepared by the double decomposition of concentrated solutions of sodium perchlorate and ammonium nitrate at 140°. If less concentrated solutions are used a lower temperature will suffice.

S. BINNING.

Explosive compositions for blasting fuses. J. P. NIENHUIS (B.P. 297,365, 17.3.27).—A slow-burning fuse powder contains saltpetre, sulphur, and oil pitch or asphalt pitch, with or without charcoal. A suitable composition is saltpetre 73%, sulphur 15%, and pitch 12%.

S. BINNING.

Cleansing agent for fire-arms. J. WAGNER (B.P. 296,550, 29.8.27).—A mixture of esters of hydroxy-fatty acids, particularly ethyl lactate, with cleansing agents known to remove acid residues will remove both acid and lead residues.

S. BINNING.

Manufacture of detonators for blasting and industrial purposes and for shells, mines, etc. NOBEL'S EXPLOSIVES CO., LTD., and G. MORRIS (B.P. 297,853, 1.4.27).—The main charge is loaded in a molten condition into the detonation casing and allowed to set, or it may be loaded as a powder and then melted. The main charge may be a single substance, *e.g.*, erythritol tetranitrate, or a mixture of at least two substances, *e.g.*, tetryl and trinitrotoluene.

S. BINNING.

XXIII.—SANITATION; WATER PURIFICATION.

Method of action of silica dust in the lungs., P. HEFFERNAN and A. T. GREEN (J. Ind. Hygiene, 1928, 10, 272—278).—Pulmonary silicosis is not due to direct injury by sharp insoluble particles, nor to the toxic action of the silica ion, but rather to the colloidal action of hydrated silica formed from minute particles of silica dust. The activity of colloidal silica in the lungs may be prevented by such substances as clay, carbon, shale dust, etc., which form a protective coating on the silica dust and coagulate any colloidal silica formed.

F. R. ENNOS.

Determination of dust inhaled by workmen. A. I. BURSTEIN (J. Ind. Hygiene, 1928, 10, 279—291).—The amount of dust drawn into any type of air filter depends on the negative pressure developed at the intake of the filter, and on the direction and position of the intake; it is therefore not necessarily an accurate measure of the amount of dust per unit volume of air in a room. The term "dust coefficient" is adopted to signify the average amount of dust inhaled by a workman during 1 hr. in a workroom. Two types of apparatus for determination of the "dust coefficient" are described: (a) the koniogravimeter, in which air is inspired through the filter by the workman, and (b) consisting of a pump and bellows suitably designed to imitate the process of human respiration.

F. R. ENNOS.

Treatment of boiler-feed waters of low incrustant content. S. C. JOHNSON (Ind. Eng. Chem., 1928, 20, 1071—1072).—It has been found that in locomotive practice the use of soda ash and/or sodium aluminate is advantageous even with feed waters of 34—86 p.p.m. of total hardness. Systematic blowing down is also essential.

C. IRWIN.

Zeolite softening of lime-treated water at Columbus, Ohio, Water Softening and Purification Works. C. P. HOOVER, V. L. HANSLEY, and C. Q. SHEELY (Ind. Eng. Chem., 1928, 20, 1102—1105).—An investigation was made of the operating costs of zeolite softening in conjunction with lime treatment to remove temporary hardness, in place of the use of soda ash. Two small-scale softeners were used, one charged with greensand and one with a synthetic zeolite, and the following data were obtained. Greensand removed 2500 grains of hardness per cub. ft. between regenerations, the synthetic zeolite 5000 grains. Salt consumption was 0.337 lb. per 1000 grains of hardness. It is more economical to use too little salt than too much. Water losses were 2.4—4.6%. Cost analyses

showed a considerable saving by the use of zeolite, but no account was taken of loss of material by disintegration. The water used contained 70—100 p.p.m. of permanent hardness.

C. IRWIN.

Road-surfacing materials. BUTTERFIELD.—See IX. Toxicity of lacquer solvents. SMYTH and SMYTH, JUN.—See XIII.

PATENTS.

Treatment of garbage etc. R. P. KINKEL (U.S.P. 1,684,448, 18.9.28. Appl., 21.3.25).—Garbage, after being dried in a current of hot gases with recovery of ammonia, is utilised as a source of heat for the continuance of the process.

C. JEFSON.

Manufacture of a disinfectant. KONIGSBERGER ZELLSTOFF-FABR. U. CHEM. WERKE KOHOLYT A.-G. (G.P. 448,705, 2.8.24).—The product (26% Cl) from chlorination of sulphite-cellulose liquor is mixed with an equal weight of unchlorinated liquor, and after a short time forms a solution which can be diluted with water. The chlorinated compound may also be compressed with an equal weight of sulphite-cellulose pitch into soluble pastilles.

C. HOLLINS.

Sterilising water. O. and R. ADLER (B.P. 292,099, 31.5.28. Czechoslov., 13.6.27. Addn. to B.P. 286,338; B., 1928, 350).—Iron, manganese, and other heavy-metal salts are removed in the prior process by passing the water, before it is treated with material containing carbon, over insoluble material resistant to the action of free or active chlorine and capable of precipitating the salts, *e.g.*, manganese dioxide, ferric oxide, etc., alone or mixed with acid-binding agents, *e.g.*, limestone, chalk, bauxite, etc. The material may also serve to obstruct the passage of the bacteria, or may be mixed with the material used for this purpose.

L. A. COLES.

Preparation of a scale-removing and -preventing substance for use in steam boilers. SOC. ANON. SUBOX (B.P. 281,598, 20.5.27. Fr., 4.12.26).—A natural resin, *e.g.*, colophony, is saponified with hot dilute sodium carbonate solution, and hydrochloric acid is added to the cooled solution to precipitate the resin acids in a hydrated state. After excess of water has been removed by pressure, protective colloids, *e.g.*, gum arabic, tragacanth, may be added to the paste-like product together with tannin, dextrin, etc. if desired.

W. G. CAREY.

Increasing the efficiency of zeolites in treatment of water. W. C. FOSTER and A. C. GARRETT (U.S.P. 1,683,967, 11.9.28. Appl., 17.1.24).—Water having low alkalinity is treated with an alkaline salt and, before the alkalinity of the water diminishes, is passed through the zeolitic bed.

F. G. CLARKE.

Filtration of alkaline waters. R. E. HALL, Assr. to J. M. HORWOOD (U.S.P. 1,686,558, 9.10.28. Appl., 3.5.24).—See B.P. 291,970; B., 1928, 626.

Compounds from indene and phenols (B.P. 297,075). Disinfectants (B.P. 297,074). Amines of hydroaromatic-aliphatic series (B.P. 297,484).—See III.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DEC. 7, 1928.

I.—GENERAL; PLANT; MACHINERY.

Pulverising hard substances. H. VON WARTENBERG [with B. STRZELCZYK and G. BORRIS] (Chem. Fabr., 1928, 617—619).—The degree of contamination of samples of alumina, fused zirconia, quartz, and glass after grinding to an "impalpable" powder in mortars of various materials has been determined. Least contamination is obtained in mortars of "Widia" metal (tungsten carbide in cobalt), but thickly chromium-plated steel and 12% manganese steel behave almost as well and have the advantage that the contamination is readily removed by treating the comminuted sample with hydrochloric acid. Agate mortars lost twice as much and porcelain mortars six times as much in weight as manganese steel mortars. Hammering in "diamond-steel" mortars of non-splitting manganese steel, using heavy blows, will reduce up to 30% of the sample through a screen with 0.1 mm. holes without introducing any appreciable contamination.

A. R. POWELL.

Efficiency and economics of so-called "colloid mills." F. HEBLER (Chem. Fabr., 1928, 581—582).—Experiments are recorded which show that colloid mills of the type of the Plauson mill are very inefficient both as regards fineness of the product and power consumption. On the average only about 25% of the substance is comminuted into particles having a diameter smaller than 100μ in 30 min., and this percentage increases only very slowly with more prolonged treatment. The power consumption to obtain 25% smaller than 100μ in 30 min. is of the order of 80 kw.-hr./100 kg., and to obtain 30% smaller than 100μ in 45 min. it is about 275 kw.-hr./100 kg.

A. R. POWELL.

Filtration apparatus for washing nickel catalyst. Y. SOSENSKI (Oil Fat Ind. Russia, 1926, No. 7—8, 68—70).—An open container is provided with a discharge pipe and valve leading to a second container. Close to the bottom of the first container are two perforated plates carrying a filtering cloth, the catalyst and solution above the plates being mechanically agitated. The drum-container, provided with a level-gauge, is connected to a vacuum pump and discharges into a third container (Russ. Pat. 2364 of 1925). CHEMICAL ABSTRACTS.

Prevention of frothing during vacuum evaporation. R. KUMMER (Pharm. Ztg., 1928, 73, 1246—1248).—Two forms of apparatus are described for the evaporation of froth-forming solutions under reduced pressure. The principle used in each case is the repeated destruction of the froth by admitting periodically a small volume of air into the heating vessel. In one modification this is effected by means of a motor-driven eccentric disc which opens and closes a valve through which a

regulated volume of air is admitted to the heating vessel. The regulator in the other modification comprises a mercury valve consisting of two tubes, one inside the other, terminating at their upper ends in splash traps, the lower end of the outer tube being closed and filled with mercury into which the lower end of the inner tube dips. The upper parts of the traps are connected to one another by means of a horizontal tube carrying a stopcock. The whole is arranged as a manometer to the distillation vessel so that the pressure in the heating vessel may be maintained at any desired value.

A. R. POWELL.

The Cottrell-Moeller process [for precipitation of dust from gases]. P. VER ECKE (Bull. Féd. Ind. Chim. Belg., 1928, 7, 293—305, 341—364).—The theory and history of the precipitation of mist and dust by the electrostatic field is summarised. There are three main types of precipitator: (A) That in which the electrodes are respectively a metallic tube and a wire hung axially. This is heavy, but permits of slow gas speeds and a maximum potential difference as the field is uniform. It is important in the metallurgy of non-ferrous metals and in blast-furnace gas purification. (B) The type with curved sheets arranged between the discharging points. This is lighter, permits of higher gas velocity, and gives less current loss, but the purification is not so good. The later forms provide for a horizontal gas flow and vertical collecting surfaces. (C) Type consisting of screens of fine wire netting. This is cheap and light, but only gives moderate yields. It is suitable for the recovery of dusts of low value from large gas volumes. The degree of purity required for any gas is determined by economic considerations. Fractional precipitation may be arranged. The transformers usually work at 50,000—60,000 volts, but should be capable of resisting a 50—100% overload in case of short-circuits in the precipitator. The types of transformers and insulation problems are discussed. The power consumption of a Cottrell plant is 3—35 kw./1000 m.³ of gas. In the cement industry the Cottrell process has rendered recovery of potash possible. For complete volatilisation a temperature of at least 1100° is necessary. Chlorides accelerate volatilisation, but sulphates retard it. From 59% to 98% of the potash is recovered in a soluble form. In some cases it has been profitable to increase the output of potash by adding felspar. In metallurgy Cottrell plants have application in the recovery of dusts, in the drying of slimes, and in the recovery of arsenic, copper, tin, mercury, lead, and zinc from roaster gases. These and other metals are also recovered from the gases from converters, cupelling furnaces, from the preparation of alloys, etc. A Cottrell plant serving for the lead, copper,

zinc, and other dusts from a Belgian metal works is described in detail. It treats 20 m.³ of gas per sec. at 70° containing 2–3 g. of dust per m.³. The gases are humidified to 30–40%. C. IRWIN.

Carbon dioxide foam in fire extinguishers. J. BRANDL (Kolloid-Z., 1928, 46, 145–148).—A discussion on fire extinguishers. E. S. HEDGES.

See also A., Nov., 1180, **Thermal conductivity of gas mixtures** (GRÜSS and SCHMICK). **Determination of concentration of solutions** (JAEGER). 1183, **Permeability of colloidal substances to gases** (KANATA). 1190, **Distillation of liquid mixtures** (TSUKAMOTO). 1195, **Crystallisation velocity in binary mixtures** (RAWITZER). 1205, **Micro-elementary analysis** (GOUBAU). 1208, **Pipette viscosimeter** (FERRIS). **Resistance thermometer** (MICHELS and GEELS). **Thermostat** (NOYES).

Tintometer. PARKER.—See XV.

PATENTS.

Furnaces for steam boilers, retorts, etc. R. HICKTON, W. W. BAGGULEY, G. BIRKS, and R. G. HICKTON (B.P. 297,395, 18.3.27).—The air for combustion is preheated in the hollow walls of the furnace, the bricks are so laid that there is no straight-through joint through which gases might percolate through the walls. A suitable form of brick or block is described in B.P. 296,033. B. M. VENABLES.

Heat exchangers. COMP. DES SURCHAUFFEURS (B.P. 292,070, 5.6.28. Fr., 11.6.27).—In a heat exchanger with vertical flow, the outer fluid is exhausted through a siphon pipe with substantially equal legs, the height of the whole siphon being adjustable so that more or less of the heat-interchanging surface is effective. The position of the siphon may be controlled by a thermostat. B. M. VENABLES.

Heat exchangers S. ROBSON (B.P. 297,509, 28.6.27).—A heat exchanger of the bundle of tubes type is made suitable for gases which may deposit corrosive liquid (e.g., sulphur dioxide or trioxide in the "contact" sulphuric acid process) by arranging the tubes vertically and dishing the tube plates so that any liquid can be drained away through pipes provided from the outside of the upper tube plate and from the inside of the lower. The side entry and outlet for the outer fluid is also arranged as a trunk surrounding a large arc of the shell with numerous inlets thereinto, giving an even distribution of the gas. B. M. VENABLES.

Heat exchangers. J. HEIZMANN (B.P. 297,613, 7.3.28).—A heat exchanger is constructed in the form of a column built up to any height from interchangeable units, which permit one fluid to flow horizontally and the other vertically. Different forms of connecting passages and baffles are provided, so that the horizontally-flowing fluid may pass many times through paths of narrow cross-section or fewer times through those of larger cross-section. B. M. VENABLES.

Heat-exchange or sterilising apparatus. R. SELIGMAN (B.P. 297,589, 3.11.27).—In heat exchangers or sterilisers consisting of a number of plates assembled as in a filter press, to cause the liquid to pass over the

plates in a uniform layer, a groove is formed in the inlet side of the plate, which gets shallower as it extends across the edge of the plate from the inlet port, and at the outlet side is another groove, which gets deeper in the same direction towards the outlet port at the corner opposite to the inlet. The grooves may have ridges of varying height alongside them.

B. M. VENABLES.

Grinding mills. MACAO-WALZENMÜHLENGES. M.B.H. (B.P. 291,803, 9.6.28. Ger., 9.6.27).—A number of planetary grinding discs rotate against a plane disc, the planetary discs are preferably eccentric to the axes of their gear wheels, and may be oval or other shape not circular. The disc mill may be combined with a planetary roller mill, the latter performing some of the grinding and forcing the material into the former.

B. M. VENABLES.

Whirling mill for the production of finely-divided powder. E. PODSZUS, Assr. to HARTSTOFF-METALL A.-G. (HAMETAG) (U.S.P. 1,685,956, 2.10.28. Appl., 8.10.23. Ger., 21.9.23).—Within a casing which is normally closed, two propellers facing each other revolve in opposite directions producing currents of air and powder which thrust against each other while whirling. Recesses are formed in the walls of the casing behind the propellers.

B. M. VENABLES.

Elutriators for testing finely-divided, pulverulent, or like materials. L. ANDREWS (B.P. 297,369, 14.6.27).—An elutriator, arranged for rapid working and with provision for subjecting the inflowing pulp to impact to break up agglomerations, is described.

B. M. VENABLES.

Producing homogeneous mixtures of pulverulent materials. M. VOGEL-JORGENSEN (B.P. 273,768, 4.7.27. Denm., 2.7.26).—The production of a perfect mixture of dry materials of constant composition though the constituents may vary is effected as follows. Each constituent is "homogenised" separately by aeration and stirring, then proportions of each to give the required final composition are mixed together by similar means. The final homogenising is performed in one of the vessels in which one of the constituents was homogenised, and may be done before the final grinding. The method is suitable for the production of cement by the dry process. [Stat. ref.] B. M. VENABLES.

Separation of solid materials of different sp. gr. G. RAW (B.P. 297,757, 26.5.27).—The material is stratified by the action of air coming up through a pervious shaking table, and flows in a longitudinal path which is alternately parallel and tapering. At the constrictions or tapered portions the deck has a steeper slope and should not be perforated for air currents. Skimmers for removing the upper strata are provided in the parallel zones just before the constrictions.

B. M. VENABLES.

Separation of dry materials. H. M. SUTTON and W. L. and E. G. STEELE (B.P. 297,876, 30.6.27).—Various forms of a shaking table with deck pervious to air currents and provided with riffles are described.

B. M. VENABLES.

Mixing solids with liquids. W. EVANS, Assr. to NAT. ALUMINATE CORP. (U.S.P. 1,686,076, 2.10.28.

Appl., 6.6.27).—A continuous stream of pulverised solids is projected at high speed against the surface of the liquid with which it is to be mixed.

H. ROYAL-DAWSON.

Stabilisation of disperse systems with coherent liquid phases. H. RINDERKNECHT (Swiss P. 121,097, 4.12.25).—Systems such as a clay soil are converted into a reversible product by the addition of salts of organic acids of high mol. wt., *e.g.*, weakly ammoniacal "noriganth" mucilage, followed by an organic acid capable of decomposing the previously added salt, *e.g.*, oxalic acid. A gelatinous product is obtained which can be liquefied by mechanical shock. L. A. COLES.

Pulp-straining machines. J. WHITE (B.P. 297,649, 31.3.28).—In a machine of the type where a pervious drum rotates in a bath of the pulp and a diaphragm is reciprocated near the surface of the drum, the diaphragm is extended from the usual quarter circumference to embrace three quarters of the circumference in three sections, the middle and lower ones being reciprocated as usual and causing the upper sections to swing so that all parts of the diaphragm move in an approximately radial, and therefore efficient, path.

B. M. VENABLES.

Thickening apparatus [for hot pulp]. H. S. COE (U.S.P. 1,686,203, 2.10.28. Appl., 9.1.25).—The feed flows over a horizontal baffle down the outside of a "heat-transferring wall" or cylindrical container suspended within the tank, and the clear overflow is withdrawn from the interior of the heat-transferring wall.

B. M. VENABLES.

Presses for the treatment of moist materials. F. KRUPP GRUSONWERK A.-G. (B.P. 284,694, 20.1.28. Ger., 5.2.27).—In an extraction press of the type in which a worm presses the material through a passage of diminishing size, the passage is formed in alternate parallel and tapering zones and the blades of the worm are omitted in the tapering zones. B. M. VENABLES.

Continuous countercurrent treatment [filtration] of liquids and solids. F. W. MANNING (U.S.P. 1,686,092—4, 2.10.28. Appl., [A] 20.3.26, [B] 11.4.27, [C] 21.6.27).—In (A) a treating solid is added to the liquid and the liquid filtered through the solid. In (B) and (C) the filter surface is continuously renewed by unwinding a strip of fabric. B. M. VENABLES.

Filter. H. WITTENBERG (G.P. 450,388, 15.7.26).—In a continuously operated filter, sludge is forced forward without turbulence into a channel formed between more or less parallel porous plates, and is washed by percolation of washing fluid through the mass.

J. S. G. THOMAS.

Apparatus for mixing and spraying liquids. F. E. SMITH, A. P. H. DESBOROUGH, W. T. THOMSON, H. F. REYNOLDS, and E. W. BLAIR (B.P. 297,558, 26.8.27).—The liquids are admitted separately into an outer tube and pass up to the closed end within which a whirling cup is situated; the liquids pass tangentially into the cup and out downwards through an inner tube.

B. M. VENABLES.

Filters or separators [for liquids]. Apparatus for separating liquids. B. D. COMYN and L. ROTTENBURG (B.P. 247,936 and 297,551, [A] 25.8.27, [B]

10.8.27).—(A) An emulsion of liquids, *e.g.*, oil and water, is passed through screens of filamentary material, at least partly composed of wire, the disperse phase being thereby caused to coalesce, and the two liquids are then separated by gravity, *e.g.*, in apparatus as described in (B) in which the mixture of liquids of different density is fed to the axis of a vertical cylindrical tank and passes outwards between spiral guides without eddies or oscillation and with gradually decreasing velocity, the separated lighter and heavier liquids being drawn off near the periphery through adjustable outlets at the top and bottom respectively. B. M. VENABLES.

Centrifugal separators. SUNDERLAND FORGE & ENGINEERING Co., LTD., and G. BLAIR (B.P. 297,914, 26.7.27 and 11.1.28).—A centrifugal separator which operates by density has a cylindro-conical bowl and horizontal axis; the pulp is fed well into the wider cylindrical end by means of a distributor, and does not pass through a hollow shaft. The separated liquor overflows at the wide end over an adjustable lip, whilst the solids are caused to travel in the opposite direction by means of an internal worm rotating at a speed slightly different from that of the bowl. B. M. VENABLES.

Centrifugal separators. AKTIEBOLAGET SEPARATOR (B.P. 292,594, 11.6.28. Swed., 23.6.27).—In a separator provided with a number of conical discs, the feed holes are formed through the inner non-conical flanges bounding the discs, and the caulks for separating the discs are also between the flanges. B. M. VENABLES.

Hydro-extractors and like centrifugal machines. K. F. G. VENZKE (B.P. 297,616, 5.1.28).—In a centrifugal machine having a horizontal shaft, a perforated basket, and mechanical means for continuously removing the collected solids, baffles and guides are provided to prevent flow of liquid into the wrong outlet of the machine. B. M. VENABLES.

Horizontal evaporator. E. SCHLEGEL (G.P. 446,659, 13.6.22).—Horizontal boiling tubes, partly filled with liquid, are arranged in sequence above one another, and liquid to be evaporated overflows from the tubes over hoods placed in the tubes below. J. S. G. THOMAS.

Purifying air or other gases. W. H. TAYLOR and C. SHAW (B.P. 297,510, 29.6.27).—Air or other gas leaving air compressors or other blowers is caused to change its direction several times and finally passes through a fabric filter. B. M. VENABLES.

Extraction of dust from gas. T. THOMSON and N. NISBET (B.P. 297,543, 23.7.27).—The stream of dust-laden air enters a first separator tangentially to produce a whirling motion, and a first portion of clean air is withdrawn from the axis of that separator. The dust and some air pass downwards and circumferentially into a second separator provided with baffles, where the dust is finally collected and the remaining air is withdrawn, preferably by a fan which returns it to the original ingoing stream of air. B. M. VENABLES.

Viscosimeter. J. WOUDEHUYSEN and G. ORY (U.S.P. 1,685,896, 2.10.28. Appl., 7.7.27).—The liquid to be tested is placed in a cup-like container in the bottom of which is a nozzle having an internal valve seating co-operating with a valve that forms part of a tubular

fitting containing a thermometer, the bulb of which dips into the liquid. Means are provided for opening and closing the valves and for collecting the liquid which flows through the nozzle. H. ROYAL-DAWSON.

Apparatus for carrying out chemical reactions between gases, or vapours, or gases and vapours. H. HARTER (B.P. 298,108, 1.7.27).—See U.S.P. 1,678,778; B., 1928, 697.

[Heating apparatus for] burning of [atomised] liquid fuel. J. N. WETHERILT and T. G. WILLIAMS (B.P. 298,146, 4.7.27).

Manufacture of containers, or capsules, for liquids, or gases, under pressure. R. H. CAMPBELL, and SPARKLETS, LTD. (B.P. 298,278, 11.7.27).

Oil from oil-laden gas (B.P. 297,622).—See II. **Heat-insulating sheets** (B.P. 274,471).—See IX. **Gas purification** (G.P. 448,034, 447,912, 450,391, 448,505, 450,392, and 448,691).—See XI.

II.—FUEL; GAS; TAR; MINERAL OILS.

Composition of coal. Oil-yielding constituents. R. HOLROYD and R. V. WHEELER (J.C.S., 1928, 2669—2677).—Results of coal distillation experiments are correlated with those of solvent analysis, using a bright coal from the Wigan Six-Foot seam. 250 g. of coal were slowly heated *in vacuo* at successive temperatures from 100° to 550°, in an apparatus similar to that used by Burgess and Wheeler (cf. B., 1914, 191), and the products were collected and examined. Hydrocarbons and resins extractable from the coal by organic solvents yield a high proportion of the oils obtained on distillation, the former being distilled unchanged quantitatively and the latter partly distilled and partly decomposed. The coal has a definite thermochemical decomposition point (318°) unaltered by changes in experimental conditions. This decomposition (of the ulmin constituents) yields water, phenolic substances, and hydrocarbon oils, and once the decomposition point has been passed the character of the coal, as shown by its behaviour towards solvents, its caking power, and reactivity, undergoes a sudden and marked change. These conclusions are confirmed by the examination of a number of bituminous coals. C. B. MARSON.

Chemical constitution of peat. I. Chemical nature of organic complexes in peat and methods of analysis. S. A. WAKSMAN and K. R. STEVENS (Soil Sci., 1928, 26, 113—137).—A division into the fractions ether-soluble, soluble in hot and in cold water, alcohol-soluble, hemicelluloses, pentosan, celluloses, lignin, crude protein, and ash accounts for about 90% of the peat and serves to distinguish between peats of different origin. A comparison of different peats and of the plants responsible for their formation is made, and the differences are discussed. H. J. G. HINES.

Heat of combustion of foundry coke and other varieties of carbon. W. A. ROTH (Arch. Eisenhüttenw., 1928—9, 2, 245—247; Stahl u. Eisen, 1928, 48, 1442—1443).—The calorific value of coke containing 1% S calculated on an ash- and moisture-free basis is 7966 ± 10 g.-cal./g. The calorific value of α -graphite, d 2.28, formed at low temperatures under high pressure, is

7832 ± 2 g.-cal./g., that of β -graphite, d 2.22, formed at high temperatures under low pressure, is 7856 ± 2 g.-cal./g., and that of "amorphous" carbon, d 1.86, is 8148 g.-cal./g. The last-named becomes denser and the calorific value becomes lower after heating above 1000° owing to slow transformation into graphite taking place. Carbon deposited from acetylene at a high temperature is almost pure graphite with a calorific value of 7886 g.-cal./g., whereas that deposited on a cold metal plate held in the acetylene flame has a calorific value of 8130 g.-cal./g. Carbon from methane and from carbon monoxide is β -graphite, whereas "temper" carbon is α -graphite. A. R. POWELL.

Bunsen flames of unusual structure. F. A. SMITH and S. F. PICKERING (Ind. Eng. Chem., 1928, 20, 1012—1013).—Mixtures of air or oxygen with propane or acetylene were burnt and the structures of the flame observed. Using acetylene-air mixtures several types were observed, viz., four distinct combustion zones, two zones which merged, and sometimes flame with a hollow dark core. When secondary air was excluded the primary combustion surface of propane-air mixtures became polyhedral and, depending on their composition, could be made to rotate or remain stationary. The number of sides is a function of the burner size and the gaseous composition. Primary zones of certain propane-oxygen mixtures, burning in secondary air, were polyhedral, with luminous streamers rising from the tip and corners of the flame. Mixtures containing more than 33% O₂ caused the flame to rotate, the speed of rotation increasing with oxygen content. Photographs of the flames described above are given. C. B. MARSON.

Diffusion flames. S. P. BURKE and T. E. W. SCHUMANN (Ind. Eng. Chem., 1928, 20, 998—1004).—The term "diffusion" flames is applied to flames in which the combustible gas is not mixed with air prior to combustion. The theory is advanced that in these flames the combustion is controlled by the rate at which the gas and air mix by diffusion. An expression is derived theoretically, based on certain assumptions, which enables the physical characteristics of "diffusion" flames to be calculated. In an experimental investigation the flames were produced by passing gas up an inner duct with a definite velocity and burning it in a mixture of oxygen and nitrogen moving up an outer duct with the same velocity. By varying the composition of the mixture the gas was burnt in either an insufficiency (underventilated) or an excess (overventilated) of oxygen. The results obtained by varying different factors individually supported the proposed theory. Keeping the flow of gas and air constant, the height of the flame varied directly as the widths of the ducts for flat flames, but cylindrical flames are not affected. The height varies with the coefficient of diffusion and directly as the velocity of flow. The effect of adding an inert gas or a small amount of oxygen to the combustible gas is to increase the height of underventilated, and diminish that of overventilated, flames. The larger the quantity of oxygen required by a gas for its combustion the taller is the flame if overventilated, and the shorter if underventilated. The

effect of varying the pressure and of preheating the gas and air was small. A method is given for deriving the coefficients of diffusion from analyses of samples of gas taken at points along the axis of the flame.

F. J. DENT.

Flicker of luminous flames. D. S. CHAMBERLIN and A. ROSE (Ind. Eng. Chem., 1928, 20, 1013—1016).—The luminous flames of various gases have been filmed. The gas was supplied at constant pressure and known rate to a burner, fitted with a lava tip and having no air ports, situated in a cubical asbestos box provided with a quartz window and an adjustable top opening by means of which the draught was regulated. The flames were photographed, at a distance of 2.29 m., with a moving-picture camera allowing of 32 exposures per sec. Hydrogen, carbon monoxide, and hydrogen sulphide flames were not sufficiently actinic to affect the film under these conditions. The films obtained with the flames of natural gas, ethylene, methyl chloride, ethane, and butane, when developed, revealed a series of flame images which identifies the flicker with an up-and-down motion confined to the upper and middle portions of the flame, and shows the downward movement to be extremely rapid. The lower portion of the flame remains quite steady, but periodically emits another flame which, though of short existence, rises above the main flame. The rate of vibration, flame speed, and amplitude of vibration were determined under different conditions. The rate of vibration for the flames of all the gases investigated was of the order of 10 per sec., and was almost independent of the rate of flow of gas, the atmosphere in which the flame burns, the tip of the burner, and the magnitude of the draught.

A. WILSON.

Radiant energy from flames. W. E. GARNER (Ind. Eng. Chem., 1928, 20, 1008—1012; cf. Johnson, A., 1928, 353).—A discussion of the thermal and chemiluminescence theories of radiant energy from flame leads to the conclusion that the emission is very largely chemiluminescence. The effects of the addition of oxygen, nitrogen, and hydrogen on the emissivity from carbon monoxide and oxygen have been investigated. The apparatus used has been described by Garner and Johnson (A., 1928, 375). Oxygen and nitrogen, while decreasing the flame temperature, increase the emissivity; this evidence supports the above conclusion. Hydrogen (2%), while increasing the temperature and accelerating the speed of the flame, diminishes the radiation sevenfold. This conservation of chemical energy within the flame is termed "energo-thermic catalysis." Measurements of the total radiation from, and the speeds of, the carbon monoxide flame show that the decrease in emissivity on the addition of hydrogen takes place in a stepwise manner, the step occurring in the vicinity of 0.03% H_2 (cf. Garner and Roffey, A., 1928, 105). It is concluded that hydrogen acts as a catalyst in the chemical sense when its percentage exceeds 0.02, whereas the energo-thermic effect is present throughout the range of concentrations up to 2%. In the latter case the deactivator is either the proton or the electron; collisions between the products of combustion and protons or electrons conserve the chemical energy within the flame.

A. WILSON.

Measurement of the temperature of stationary flames. A. G. LOOMIS and G. ST. J. PERROTT (Ind. Eng. Chem., 1928, 20, 1004—1008).—The concept of temperature as applied to flames is discussed. Proposed methods for measuring the temperature of flames are critically reviewed. Methods which depend on the immersion of thermo-couples or wires into flames cannot be used above 1800°, and the possibility of catalytic action makes their results uncertain. The method of Kurlbaum and Féry depending on spectral-line reversal is described and examined in detail. It was adopted by the authors as being the most free from theoretical objections, and the experimental manipulations involved are made easily and rapidly. The flame temperatures of Pittsburg natural gas, methane, propane, and carbon monoxide have been measured by this method for various gas-air ratios. The maximum temperatures observed were 1875°, 1878°, 1935°, and 1960°, respectively. The difference between the observed and the calculated values is explained by radiation from the flame and possible inaccuracy of the specific heat data.

F. J. DENT.

Some flame characteristics of motor fuels. G. B. MAXWELL and R. V. WHEELER (Ind. Eng. Chem., 1928, 20, 1041—1044; cf. B., 1928, 511).—In order to ascertain the cause of "pink" or "knock" of motor fuels, a photographic study has been made of the movement of flames, simultaneously with measurements of the development of pressure during the explosion of a charge within a stainless-steel cylinder. Explosions of mixtures of pentane and air, and benzene and air, at various temperatures and pressures, and various blended mixtures of benzene and pentane, and of ethyl ether with pentane, have been studied. In a pinking explosion, *e.g.*, pentane-air mixtures at high initial pressures, combustion is not complete in the flame front, but some additional impetus, such as impact of the flame with the end of the cylinder, appears necessary to cause completion of the reactions, which then take place almost instantaneously with a sudden increase of pressure. Combustion reactions are complete behind the flame front in non-pinking explosions such as those of benzene-air mixtures. The effects of length and shape of cylinder and also the effects of turbulence and the addition of anti-knock compounds are discussed. The action of lead tetraethyl seems to depend on the decomposition products of the compound rather than on the compound itself.

G. CLAXTON.

Relative rates of reaction of olefines in combustion with oxygen and in oxidation with aqueous potassium permanganate. H. S. DAVIS (Ind. Eng. Chem., 1928, 20, 1035—1057).—Various mixtures of ethylene and propylene and ethylene and isobutylene were exploded with oxygen and the resulting gases were analysed. In all experiments the ethylene was less readily oxidised than either propylene or isobutylene. Solutions of ethylene and isobutylene in water were oxidised at room temperature with a deficiency of potassium permanganate. After oxidation the dissolved gases were boiled off and analysed. As before the ethylene was less readily oxidised. The ratios of the relative rates of reaction of ethylene, propylene, and isobutylene were: for combustion with oxygen, 1 : 1.7 :

2.8, respectively; for oxidation with aqueous permanganate, 1 : — : 1.6. Analogies between the reaction of olefines with oxygen and bromine are pointed out.

G. CLAXTON.

Action of accelerators and inhibitors upon the oxidation of liquid hydrocarbons. T. E. LAYNG and M. A. YOKER (Ind. Eng. Chem., 1928, 20, 1048—1052. Cf. Moureu, Dufraisse, and Chaux, B., 1927, 243, 512, 692; Callendar, B., 1926, 618; 1927, 272).—An apparatus has been devised and a method described for determining the effect of inhibitors and accelerators of knock on the slow oxidation of hydrocarbon fuels. Data are given to show the effect of various substances on the slow oxidation of *n*-heptane, gasoline, and kerosene at temperatures above 160°. Aniline, diphenylamine, lead tetraethyl, and potassium ethoxide all appear to be inhibitors of gas-phase oxidation, but lead tetraethyl and sodium and potassium ethoxides are accelerators of liquid-phase oxidation when present in extremely small percentages, and exhibit surprisingly similar properties. It would appear that an ideal anti-knock mixture might be obtained by incorporating a powerful inhibitor of only vapour-phase oxidation with another of only liquid-phase oxidation.

G. CLAXTON.

Importance of mixture ratio in rating fuels for knock. J. M. CAMPBELL, W. G. LOVELL, and T. A. BOYD (Ind. Eng. Chem., 1928, 20, 1045—1048).—In knock testing by means of comparisons between some standard fuel and the fuel under test, no satisfactory agreement has been observed between the results obtained using different methods for testing the same fuels. The present authors show that the tendency to knock is very sensitive to changes in the mixture strength, and the use of mixture ratios giving the maximum knock for each fuel is suggested as a convenient means for obtaining more consistent results between different laboratories.

G. CLAXTON.

Peat tar. G. STADNIKOV and N. TIROV (Brennstoff-Chem., 1928, 9, 325—326).—The low-temperature tar from a sphagnum peat has been examined. Addition of ether precipitated 6% of a black solid soluble in amyl alcohol and pyridine. Steam distillation, after evaporation of the ether, yielded 15.1% of a volatile fraction, containing 0.48% of basic and 2.4% of acidic constituents (calculated on the dry tar). By treatment of the residue with light petroleum and extraction of the precipitated pitch with benzene, 11.6% of "asphaltene" was obtained. 11.2% of solid paraffins, 13.8% of resins, 0.44% of bases, and 7.1% of tar acids were separated from the fraction soluble in light petroleum. The solid paraffins contained unsaturated compounds and possessed rather high acid and saponification values. The neutral oils consisted principally of unsaturated hydrocarbons and compounds containing oxygen.

A. B. MANNING.

Theory of the formation of petroleum. II. Composition of the low-temperature tar from Mataganski boghead coal. G. STADNIKOV and Z. VOSSCHINSKAJA (Brennstoff-Chem., 1928, 9, 326—328; cf. B., 1928, 735).—All the tar fractions contain considerable quantities of oxygen compounds. On

oxidation by permanganate they yield principally monobasic aliphatic acids. The highest-boiling fractions contain appreciable quantities of free acids and anhydrides. It is concluded that Mataganski boghead coal, like that of Tscherechovski, consists of polymerised fatty acids.

A. B. MANNING.

Treatment of Baku lubricating oil distillates by German plant. K. KOSTRIN (Azerbeid. Neft. Choz., 1928, No. 5, 55—57).—Details of treatment of solar oils with sulphuric acid, sodium hydroxide, and water for the production of transformer oils, medicinal white oil, turbine oils, machine oil, cylinder oils, and spindle oils are recorded.

CHEMICAL ABSTRACTS.

Determination of aromatic hydrocarbons in petrols by means of the "alcohol value." K. R. DIETRICH (Auto-Technik, 1927, 16, 7—9; Chem. Zentr., 1927, II, 2528).—The aromatic hydrocarbon content of a petrol can be obtained with sufficient accuracy for practical purposes from its "alcohol value," i.e., the critical dissolution temperature of a mixture of 7 pts. (by wt.) of the petrol with 3 pts. of 96% alcohol. The method cannot be applied to petrols containing an abnormally high content of unsaturated hydrocarbons, but is not appreciably affected by the amounts usually present in straight-run petrols. An approximate idea of the naphthene content of the petrol can be obtained from the "alcohol curve." A relationship exists between the tendency of a petrol to "knock" and its "alcohol value."

A. B. MANNING.

See also A., Nov., 1182, **Wood charcoal as absorbent for gases** (MAGNUS, SAUTER, and KRATZ; LOWRY and BOZORTH; ZEISE). 1193, **Velocity of flame in gaseous mixtures** (YUMOTO). **Gaseous combustion at high pressures** (BONE, TOWNEND, and SCOTT). 1194, **"Explosion limits" of carbonic oxide-air mixtures** (BONE, NEWITT, and SMITH). 1210, **Petroleum and filtering earths** (NUTTING). 1211, **Cholesterol as parent of petroleum** (STEINKOPF).

Carbides. FISCHER and BANGERT.—See VII. **Structure viscosity of oils.** OSTWALD and others.—See XII.

PATENTS.

Manufacture of coal products. KOPPERS Co., Assees. of H. V. ROSE and W. H. HILL (B.P. 268,372, 25.3.27. U.S., 25.3.26).—A stable homogeneous "solution" of coal may be obtained by heating 1 pt. of coal with 1—10 pts. of water-gas tar, coal-tar oils, or cracked petroleum oils at about 300° under atmospheric pressure or slightly above. The liquid is separated from undissolved matter and the original coal may be recovered substantially freed from ash, mineral charcoal, and inorganic sulphur by removing the solvent or by precipitation by the addition of low-boiling materials such as benzols or light petroleum. The dispersion of coal in oil may be treated with liquid extraction agents, e.g., sulphur dioxide, in order to separate certain constituents, or materials may be added to react with the organic sulphur compounds and effect their removal. By using a mixture of coals differing in character, a composite coal blend may be recovered from the liquid, or by the addition of finely-divided foreign material a solid coal product containing

such material uniformly distributed throughout the mass may be obtained. Furthermore, the coal may be dispersed in a tar, and the product subjected to a sufficiently high temperature to decompose the more volatile constituents of the tar and leave a cokeable blend of coal and pitch.

H. S. GARLICK.

Distillation of [finely-divided] coal. A. F. P. PINET and A. DEBOUT (F.P. 626,895, 30.12.26).—The coal particles fall through a narrow preheating chamber, in which partial distillation takes place, into a coking chamber below.

L. A. COLES.

Distillation of fuel having a high ash content in the form of small particles or dust. W. SCHWIER (G.P. 450,460, 2.2.24).—The fuel is blown together with air through fused non-combustible and non-volatile material contained in vessels which can be tilted or rotated so that the depth of the delivery tubes below the level of the liquid can be adjusted as required.

L. A. COLES.

Fractionation of coal, oil, and other hydrocarbons. SOC. INTERNAT. DES COMBUSTIBLES LIQUIDES (Addn. F.P. 32,045, 1.7.26, Ger., 15.12.25, to F.P. 606,191; B., 1927, 435).—Gaseous products obtained in the prior process are cooled before releasing the pressure to cause condensation of water and benzene.

L. A. COLES.

Manufacture of montan wax. A. RIEBECK'SCHE MONTANWERKE A.-G. (B.P. 288,258, 12.3.28, Ger., 8.4.27).—After extracting the wax from dried brown coal by solvents it is separated from the solution by distillation, and solvent from the residual coal is expelled by direct steam. The heat contained in the solvent vapour and steam is utilised for drying further quantities of moist raw coal, after freeing the vapours from dust.

C. B. MARSON.

Wood carbonising plant. R. MALBAY (G.P. 446,223, 7.4.26, Fr., 15.4.25).—The retorts are divided into two groups and are heated, on the one hand, by the hot gases from a generator which pass through the retorts of the first group and then through those of the second before their utilisation in a gas engine, and, on the other hand, by the exhaust gases from the engine which likewise pass through the retorts of the first and second groups in succession. As soon as the material in the first group of retorts is carbonised the path of the gases is reversed, so that the process continues without interruption.

A. B. MANNING.

Coke oven. E. COPPÉE & CIE. (B.P. 294,561, 16.12.27, Belg., 26.7.27, Addn. to B.P. 253,887; B., 1927, 721).—The oven described in the main patent is modified by the provision of gas regenerators alternating with the air regenerators beneath the ovens, in order to permit heating of the walls with poor gas. The gas regenerators are divided into separate chambers for preheating the gas supplied to the corresponding separate groups of flues. The end groups of flues communicate with the end chambers of the air regenerators, and these in turn with the other chambers. Part of the hot gases is by-passed at the outlet of the last group of flues but one, towards the corresponding gas regenerator in order to heat the latter for the following change of working. The ovens may be heated by rich gas if

desired, ducts being provided leading directly to the inlets of each group of flues.

A. B. MANNING.

Production of activated carbon. ALGEM. NORIT MAATSCHAPPIJ (B.P. 273,761, 2.7.27, Holl., 3.7.26).—A mixture of different sized pieces of carbonaceous material is charged into a retort and is subjected to dry distillation followed by activation in a current of gas or steam. The period of activation is limited so that only the smaller pieces and the external portions of the larger pieces are highly activated. After removal from the retort the material is sifted in order to separate the smaller pieces and the remainder is subjected to a further activating process. A vertical retort of elongated cross-section is used, and provision is made for the separate removal from the retort of the vapours evolved in the preliminary drying zone.

A. B. MANNING.

Eliminating the acidity of acid activated charcoal. VER. F. CHEM. U. MET. PROD. (B.P. 285,386, 14.2.28, Czechoslov., 14.2.27).—Acid activated charcoal is treated under pressure and at above 100° with alkaline substances such as milk of lime, sodium carbonate, or caustic soda. Small quantities of lime produce a neutral charcoal immediately; larger quantities of lime or soda give an alkaline charcoal. Such lime-treated charcoal eventually becomes neutral owing to carbon dioxide in the air, but soda-treated charcoals remain permanently alkaline.

C. B. MARSON.

Adsorbent medium [carbon]. E. URBAIN (F.P. 626,488, 31.3.26).—An absorbent mass of carbon and a mineral substance, e.g., a phosphate or silicate, is produced by mixing an organic substance with a mineral substance, both finely divided, drying the mixture, carbonising at 350–400°, and then heating the product to about 1000°.

J. S. G. THOMAS.

Gas-generating plants. A. V. TULLY (B.P. 297,568, 12.9.27).—In a gas generator of the type having a superimposed retort for the carbonisation of the charge before its passage into the gasification chamber below, a central poking rod, capable of rotation and/or movement vertically, is provided in order to prevent caking of the charge. The rod may take the form of a tube, and then serves as an inlet either for a carburetting hydrocarbon or for steam.

A. B. MANNING.

Apparatus for manufacture of gas from oils. C. CHIŁOWSKY (B.P. 293,008, 20.10.27, Fr., 27.10.26, Cf. B.P. 255,423, 271,899, and 271,907; B., 1927, 805, 867).—The apparatus comprises two symmetrical units which can function alternately so that while the first unit is in use, carbon deposits produced during the manufacture of the gas in the second unit are removed by combustion in a current of air which has been heated by contact with a catalyst, such air being then used for the atomisation and partial combustion of oil in the first unit. Reversal of the current of air and alternate working of the two halves is ensured by suitable valve devices. At any convenient point steam may be added to the air injected into the apparatus or to the cloud of atomised oil.

C. B. MARSON.

Drying of fuel gases. C. COOPER, D. M. HENSHAW, and W. C. HOLMES & Co., LTD. (B.P. 298,097, 2.5.27, Cf. B.P. 248,841 and 285,936; B., 1926, 428; 1928,

325).—High-pressure town's gas which is to be dried by contact with a hygroscopic material is subjected to a higher pressure than that normally necessary, and while thus compressed is treated with the drying agent. The gas is brought into contact with a liquid dehydrating agent under conditions involving its travel in opposition to a counter-pressure due to the hydrostatic head of the liquid. The liquid hygroscopic material is withdrawn continuously or periodically for dehydration, and then returned for the treatment of further quantities of gas.

C. B. MARSON.

Removal and utilisation of hydrogen sulphide from gas mixtures containing it and simultaneous production of manganese salts. CHEM. FABR. JOHANNISTHAL G.M.B.H., Assees. of M. LÖWY (G.P. 450,230, 29.6.26).—The gases act on material containing manganese dioxide in the presence of dilute hydrochloric acid or sulphuric acid.

L. A. COLES.

Separation of oil from oil-laden gas. H. A. FRASER, W. L. PRICE, and J. HETHERINGTON (B.P. 297,622, 12.1.28).—The gas is passed through a compartment packed with short lengths of metal tube on which the oil is deposited. The oil runs down into a collecting chamber at the bottom of which is a valve controlled automatically by a float within the chamber. The apparatus is designed particularly for use with refrigerating plants, to remove entrained lubricating oil from the compressed gas and to return it to the pump.

A. B. MANNING.

Production of reducing gases. G. LECOULTRE and E. BORELS (F.P. 626,044, 9.12.26. Switz., 30.9.26).—Gas mixtures containing oxygen and, *e.g.*, carbon dioxide, nitrogen, hydrocarbons, alcohol vapour, etc. are passed through heated vessels containing wood charcoal, coke, graphite, etc.

L. A. COLES.

Extraction of hydrocarbon oils from coal. J. TRAUTMANN (B.P. 272,526, 9.6.27. Ger., 11.6.26).—The coal is distilled and the volatile products, preferably after removal of the sulphur compounds, are passed directly to a hydrogenating plant without being first condensed. Hydrogen, carbon monoxide, or water-gas, and catalysts in the form of powder or vapour may be added to the distillation products before they enter the reaction chamber. A water-gas generator, utilising the coke produced, may be made a constituent part of the retort.

A. B. MANNING.

Process for splitting hydrocarbons. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,398, 16.5.27).—Hydrocarbons containing more than four atoms of carbon in the molecule are converted into a mixture of olefines and diolefines by passage over catalysts consisting of difficultly reducible oxides or compounds at 600° or above. *E.g.*, by the passage of cyclohexane through a quartz tube containing calcium aluminate at 600° it is converted principally into ethylene and butadiene.

A. B. MANNING.

Cracking of hydrocarbons. F. A. HOWARD, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,681,899, 21.8.28. Appl., 20.3.23).—Crude oil, freed from light constituents, is distilled at 5–150 lb. pressure, tar being run off and the vapours passing through a coil at 400–500° placed in the upper part of the cracking furnace but

screened from direct and radiant heat by the feed coil. The cracked vapours enter a drum maintained at 360–460° in the highest part of the furnace, and pass thence to the rectifier.

C. HOLLINS.

Treatment [cracking] of [hydrocarbon] oil. D. G. BRANDT, Assr. to DOHERTY RES. Co. (U.S.P. 1,681,321, 21.8.28. Appl., 23.3.22).—Deposition of carbon and formation of hydrogen, methane, etc. are minimised and the yield of high-boiling gasoline is improved by applying the cracking heat by means of low-boiling cracked gases preheated to about 550°.

C. HOLLINS.

Cracking of hydrocarbon oils. H. A. GILL. From GASOLINE PRODUCTS Co., INC. (B.P. 297,949, 5.9.27).—Hydrocarbon oil is heated to cracking temperature in a heating zone and passed to a reaction chamber under sufficient pressure to effect cracking substantially in the liquid phase. The products are passed to an evaporating stage of reduced pressure where cracking and vaporisation of the oil are checked by the introduction of a cooler mass of hydrocarbon oil, either at the actual point where the pressure is reduced, or at a point after the pressure on the oil discharged from the reaction chamber has been released but before the oil has reached the stage of dephlegmation. The contained heat of the mixture permits the distilling off of the lighter products, which are subjected to a refluxing and final condensation action; the heavy unvaporised portions are withdrawn from the system.

H. S. GARLICK.

Refining of hydrocarbon oils. H. J. HALLE, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,681,638, 21.8.28. Appl., 12.5.20. Renewed 12.2.27).—The oil is distilled through a column of hot 40–55% sulphuric acid to give gasoline and sludge acids.

C. HOLLINS.

Apparatus for converting hydrocarbon oils. O. C. BREWSTER, Assr. to STANDARD OIL Co. (U.S.P. 1,681,658, 21.8.28. Appl., 20.6.25).—Oil, heated to 350° by circulation through a separately fired coil, is aerated, and when conversion by internal combustion has begun oil heated to 400° in a preheater is added through a stripping column. Vapours are collected from the stripping column and the preheater.

C. HOLLINS.

Method of utilising residue [mineral] oils containing water. A. H. MALLERY (B.P. 268,814, 1.4.27. U.S., 5.4.26).—Oil residues containing water are heated to a temperature (about 670°) which is sufficient to vaporise the oil and water and bring about combustion of the oil on admitting a sufficient quantity of air.

H. S. GARLICK.

Treatment of petroleum products. E. R. WILES (U.S.P. 1,684,426, 18.9.28. Appl., 24.4.23).—Petroleum is treated with a solvent containing about 35% of acetone and 65% of benzol; on cooling the solution amorphous paraffin of high m.p. separates out.

H. ROYAL-DAWSON.

Gas condensers. W. BLAKELEY (B.P. 298,143, 28.6.27).

Gas scrubbers. S. E. SPENCER (B.P. 298,360, 31.10.27).

Extractor gear for gas retorts. GAS LIGHT & COKE Co., and S. HOLDWAY (B.P. 298,093, 28.3.27).

Gaseous hydrocarbons (F.P. 605,839). Naphthenic compound (U.S.P. 1,681,657).—See III. Esters of silicic acid (B.P. 298,778). Lead tetraethyl (B.P. 280,169).—See VII. Road repairing (B.P. 298,812). Antiseptic for wood (F.P. 608,576).—See IX. Flotation of coal (B.P. 289,848).—See X. Detection of inflammable gas (B.P. 298,767).—See XI. Waterproof paint (U.S.P. 1,684,593). Bituminous paints (B.P. 298,256).—See XIII.

III.—ORGANIC INTERMEDIATES.

Determination of aniline by Lehmann's method. A. V. PAMFILOV and V. E. KISSELEVA (Z. anal. Chem., 1928, 75, 87—92).—Lehmann's method (Lunge and Berl, "Chem.-techn. Untersuchungsmethoden," 7th ed., I, 657) for the determination of small quantities of aniline is modified as follows: 20 c.c. of the aniline solution are treated with 20 c.c. of 10% sulphuric acid, 20 c.c. of a standard hypobromite solution, and 2—3 g. of potassium bromide. After 2—3 min. potassium iodide is added and the liberated iodine, corresponding with the excess of hypobromite, is titrated with thiosulphate.

A. R. POWELL.

See also A., Nov., 1190, System cresols-amines (PUSHIN and SLADOVIC). 1195, Velocity of esterification of amides in alcohol (TAYLOR and DAVIS). 1196, Nickel catalyst for continuous hydrogenation (SVIZUIN). 1197, Vapour-phase oxidation of furfuraldehyde (SESSIONS). 1198, Photochemical decomposition of diazo compounds (EGGERT and SCHRÖTER). 1229, Determination of carbamide (LUCK). 1233, Oxidation in the benzene series by gaseous oxygen (STEPHENS). 1234, Micro-identification of isomerides and homologues in mixtures (MAGITA). Introduction of the benzyl group into the benzene nucleus (FÖLDI). 1235, Chlorodecalin (FERRERO and FEHL-MANN). 1236, Arylamine salts of naphthalene-sulphonic acids (FORSTER, HANSON, and WATSON; FORSTER and MOSBY). Nitration of substituted tertiary aromatic amines (CLEMO and SMITH). 1237, Nitro-derivatives of dibenzylaniline (DESAI). 1238, System phenols-ammonia (BRINER and AGATHAN; BRINER and MORF). 1248, Acylation and alkylation of aromatic compounds (STADNIKOV and BARYSCHEVA). 1255, 6-Aldehydocoumarin and derived dyes (SEN and CHAKRAVARTI). 1267, Micro-determination of sulphur in organic compounds (HANAI).

Combustion of olefines. DAVIS.—See II. Cymene. ASCHAN.—See V.

PATENTS.

Production of carbon disulphide. H. SCHULZ (G.P. 450,178, 3.4.25. Addn. to G.P. 407,656; B., 1926, 823).—The charcoal is charged into the furnace through a filling compartment which can be closed at the top and bottom to prevent the entry of air. The charcoal is heated to redness in cylindrical vessels in recesses in the furnace wall, and the vessels are transferred to the filling chamber and discharged by opening them from outside the apparatus. L. A. COLES.

Manufacture of solid, liquid, and gaseous hydrocarbons. J. CAMPARDOU (F.P. 605,839, 2.2.25;

cf. A., 1927, 440).—Suitable organic compounds or their vapours are heated with carbon (wood charcoal) at 500—600° (or at lower temperatures under increased pressure) to give (a) a fuel gas containing carbon monoxide, hydrogen, and gaseous hydrocarbons; (b) liquid and solid hydrocarbons; and (c) a residue of carbon. Alcohols and ethers yield paraffins; acids, ketones, aldehydes, and carbohydrates yield olefines; phenols give alicyclic and aromatic hydrocarbons. The gaseous product may be hydrogenated to give lighting gas, or treated with steam at high temperatures to give fuel gas, carbon dioxide being removed in each case.

C. HOLLINS.

Manufacture of alcohols and liquid hydrocarbons. COMP. DE BÉTHUNE (F.P. 613,541, 27.7.25).—Coal gas, or a suitable mixture of hydrogen, methane, and carbon monoxide, is led at 400—600° under 400 atm. over magnetic iron oxide, and the gaseous products, after condensation of alcohols etc., are converted into oily hydrocarbons, b.p. below 100°, by passage over active carbon at 100—400°.

C. HOLLINS.

Synthesis of methanol [methyl alcohol]. H. H. STORCH, Assr. to ROESSLER & HASSLACHER CHEM. Co. (U.S.P. 1,681,750—1,681,753, 21.8.28. Appl., [A, B] 16.5.27, [C] 27.5.27, [D] 4.6.27).—(A) A copper catalyst for reduction of carbon monoxide to methyl alcohol is prepared by adding sodium hydroxide to a cuprammonium solution, boiling to expel ammonia, and reducing the copper oxide so formed at 150—200° with hydrogen saturated with methyl alcohol vapour. (B) An intimate mixture of reduced copper and a fluoride of calcium, strontium, barium, or magnesium is used as catalyst. (C) The copper catalyst of (A) is prepared in intimate admixture with magnesium oxide. (D) A palladium catalyst intimately mixed with a non-reducible metal oxide (e.g., of chromium, zinc, or cerium) is prepared.

C. HOLLINS.

Catalysts for synthetic methanol [methyl alcohol] production. COMMERCIAL SOLVENTS CORP., J. C. WOODRUFF, and G. BLOOMFIELD (B.P. 271,840 and Addn., B.P. 279,378, 20.4.27. U.S., [A] 26.5.26, [B] 25.10.26).—(A) Methyl alcohol, uncontaminated by by-products, is obtained from oxides of carbon and hydrogen at 130—140 atm. and 340—450° by using a catalyst comprising a difficultly reducible metal oxide (oxide of zinc, magnesium, cadmium, chromium, vanadium, or tungsten) and smaller amounts of a reducible metal oxide (oxide of copper, silver, iron, nickel, cobalt) and a metal halide (zinc chloride). E.g., a catalyst, made by dissolving 225 pts. of nickel nitrate crystals in water, adding 750 pts. of zinc chloride, precipitating nickel hydroxide with ammonia, filtering, drying, adding a solution of 148 pts. of zinc chloride, and drying, yields 2.5 litres per hr. of 56% methyl alcohol from a gas containing 8% CO₂, 3% CO, and 89% H₂ at a space velocity of 75,000—100,000. (B) An improved catalyst is made by adding an aqueous solution of chromium nitrate, ferric nitrate, and zinc chloride to a suspension of zinc oxide, and drying the paste at 350° in a current of hydrogen. At 200—220 atm. and 400—420° a space velocity of 20,000—40,000 gives a yield of 1.5—2.5 litres per hr. of 93—97% methyl alcohol. C. HOLLINS.

Manufacture of concentrated acetic acid (or a mixture of acetic acid and acetic anhydride) with simultaneous production of unsaturated hydrocarbons. HOLZVERKOHLLUNGS-IND. A.-G. (B.P. 274,076, 1.7.27. Ger., 6.7.26).—Ethyl acetate vapour is passed through a quartz tube charged with a catalyst (copper, silver, or a phosphate other than iron phosphate) at 600°; 98% yields of acetic acid (99%) and ethylene are obtained. Contact with iron compounds must be avoided. C. HOLLINS.

Production of acetic acid [from wood]. F. G. RAWLING (U.S.P. 1,681,684, 21.8.28. Appl., 12.2.26).—Wood is impregnated with sodium sulphite and enough sodium carbonate or hydrogen carbonate to combine with the acetic acid formed and prevent production of bisulphite. The wood is drained, and steamed at 110–160° in a closed vessel. The residue is used for pulping, dry distillation, etc.; the aqueous liquid is acidified and acetic acid distilled off. C. HOLLINS.

Halogenation of organic compounds. I. G. FARBENIND. A.-G. (B.P. 283,877, 18.1.28. Ger., 18.1.27).—The hydrogen halide produced during halogenations is utilised in the same or consecutive operations for the conversion of methyl alcohol into methyl halide. The process is useful in the chlorination of methane or methyl chloride, the mixed gaseous products being passed into boiling methyl alcohol or with methyl alcohol vapour over a catalyst. C. HOLLINS.

Catalytic oxidation of organic and inorganic substances. O. Y. IMRAY. From MONSANTO CHEM. WORKS (B.P. 298,142, 25.5.27).—A vanadium pentoxide complex silicate catalyst, intended especially for manufacture of sulphuric acid by the contact process, is made, for example, by adding acid to mixed solutions of potassium vanadate, silicate, and borate; the gel is pressed and dried at 100°, broken up, acidified, and, after oxidation with hydrogen peroxide, dried at 300°. Such a catalyst is unaffected by the usual catalyst poisons, and is also suitable for oxidation of toluene to benzaldehyde and benzoic acid. C. HOLLINS.

Heating of catalysts for pressure-synthesis of gas mixtures. G. L. E. PATART (F.P. 618,439, 2.7.26).—In order to secure uniform temperature the catalyst is mixed with a conducting material and heated electrically. *E.g.*, for ammonia synthesis silica, asbestos, magnesia, or alumina is added to the iron catalyst; for alcohols, acids, etc. from carbon monoxide and hydrogen copper, silver, aluminium, coal, coke, or graphite is added to the zinc oxide–chromium oxide catalyst. C. HOLLINS.

Production of aldol. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 298,349, 19.9.27).—A mixture of acetaldehyde (2 pts.) and 1.25% sodium hydroxide solution (1 pt.) is passed through successive spirals at 3°, 8°, 15°, and 20°, respectively. The alkali is then neutralised and water and acetaldehyde are removed by passage through towers at 40° and 70° under reduced pressure. Aldol suitable for reduction to butylene glycol is obtained. C. HOLLINS.

Manufacture of glycol. CHEM. FABR. KALK, G.M.B.H., and H. OERME (F.P. 612,825, 16.3.26).—Ethylene chloro-

hydrin is hydrolysed with calcium carbonate or calcium hydrogen carbonate solution and the metal removed as sulphate or phosphate. C. HOLLINS.

Preparation of alkali alcoholates [alkoxides] or of compounds containing alcoholic hydroxyl groups. C. VAN LOON (U.S.P. 1,681,600, 21.8.28. Appl., 20.7.27. Holl., 21.7.25).—A solution of glycol in concentrated aqueous sodium hydroxide (1 mol. or 2 mols.) is evaporated to dryness in a vacuum and the product is dried in a current of hydrogen under reduced pressure at 110–115°. C. HOLLINS.

Manufacture of dioxan. A. KNORR and G. STEINMIG, ASSRS. to I. G. FARBENIND. A.-G. (U.S.P. 1,681,861, 21.8.28. Appl., 6.8.26. Ger., 23.12.24).—A mixture of ethylene glycol (25 pts.) or $\beta\beta'$ -dihydroxydiethyl ether or its monoethyl ether and concentrated sulphuric acid (1 pt.) is heated, more glycol being added to replace the dioxan which distils off. C. HOLLINS.

Production of thiourea. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,999, 9.11.27).—Calcium cyanamide, ammonium hydrogen carbonate, and a solution of ammonium sulphide are stirred into water at 70°, calcium carbonate is filtered off, and the solution is evaporated after decolorisation with animal charcoal. C. HOLLINS.

Manufacture of non-dyeing thio-derivatives of phenols. I. G. FARBENIND. A.-G., and A. THAUSS (B.P. 298,280, 11.7.27. Addn. to B.P. 173,313; B., 1922, 139 A).—Phenols, other than those containing nitrogen, are heated at 180–210° with sulphur in presence of a neutral (to phenolphthalein) salt of an organic or a weak inorganic acid, *e.g.*, 1% of sodium formate, nitrite, sulphite, or hyposulphite. C. HOLLINS.

Compounds of arylenediamines. IMPERIAL CHEMICAL INDUSTRIES, LTD., and A. RILEY (B.P. 297,989, 17.10.27).—Diamines of the benzene or naphthalene series may be isolated by means of the sparingly soluble compounds of the bases with chlorides or bromides of zinc, cadmium, or mercury. *Compounds of zinc chloride with* *p*-phenylenediamine, m.p. above 250°, *p*-aminodiethylamine, m.p. 165–170°, *p*-aminodiphenylamine, m.p. 245°, *p*-aminoacetanilide, m.p. 205°, *m*-phenylenediamine, m.p. above 300°, 4-chloro-*m*-phenylenediamine, m.p. 239°, *o*-phenylenediamine, m.p. above 300°, and 1:2-naphthylenediamine; of mercuric chloride with *p*-phenylenediamine, m.p. 206–208°; and of cadmium bromide with *m*-phenylenediamine, m.p. 280°, are described. The zinc chloride compounds usually have the formula $B, \frac{1}{2}ZnCl_2$. C. HOLLINS.

Manufacture of compounds of the morpholine series. IMPERIAL CHEMICAL INDUSTRIES, LTD., J. B. PAYMAN, and H. A. PIGGOTT (B.P. 298,336, 1.9.27).—An alkylene or aralkylene halogenohydrin is warmed with *p*-toluenesulphonamide, and the product is heated in concentrated sulphuric acid at 160–200° with production first of a *p*-toluenesulphonylmorpholine which is then hydrolysed to a morpholine. Morpholine and its *C*-derivatives are thus prepared from the chlorohydrins of ethylene, propylene, and cinnamic acid (obtained from phenylglycidic acid and hydrogen chloride). C. HOLLINS.

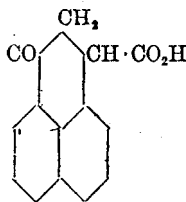
Sulphonation of aromatic compounds. L. GAY, P. MIOU, and M. AUMÉRAS (F.P. 619,436, 24.6.26).—The water formed during sulphonation is removed by entrainment in the vapour of an organic solvent which is bubbled through the hot reaction mixture. The solvent must be substantially insoluble in water and in the sulphonation mixture, and must be unattacked by sulphuric acid. C. HOLLINS.

Emulsifying agents. I. G. FARBENIND. A.-G. (Swiss P. 121,099, 23.12.25. Ger., 18.9.25).—The material contains emulsifying agents, *e.g.*, alkyl-substituted aromatic sulphonates, and colloidal material of high mol. wt., *e.g.*, glue or water-soluble rubber products. L. A. COLES.

Manufacture of 6-nitro-2-amino-1-benzoic [6-nitroanthranilic] acid. I. G. FARBENIND. A.-G. (B.P. 286,694, 8.3.28. Ger., 9.3.27. Addn. to B.P. 285,877; B., 1928, 847).—6-Nitroanthranilic acid, m.p. 184—185° (decomp.), is obtained by heating 2-chloro-6-nitrobenzoic acid with ammonia and copper at 120° in a closed vessels. C. HOLLINS.

Oil-soluble naphthenic compound. O. E. BRANSKY, Assr. to STANDARD OIL Co. (U.S.P. 1,681,657, 21.8.28. Appl., 29.10.23).—A naphthenic lubricant distillate, *e.g.*, from Louisiana oil, is neutralised with sodium carbonate solution and washed with alcohol. The washings on evaporation, acidification, and distillation in steam yield a viscous oily acid. The sodium salt is a detergent; the copper salt (green) may be applied in naphtha solution as a wood preservative. C. HOLLINS.

Manufacture of cyclic ketones. I. G. FARBENIND. A.-G. (B.P. 273,321, 24.6.27. Ger., 24.6.26).—Maleic anhydride is condensed in presence of sodium aluminium chloride etc. with naphthalene, acenaphthene, or other fused-ring compound (other than anthrones) having free adjacent *peri*-positions. There is first formed a keto-acid, which is subsequently condensed to a cyclic keto-acid; *e.g.*, naphthalene gives first β -1-naphthoyleacrylic acid, m.p. 139—140°, and then the acid, m.p. 186—187° (probably annexed formula). Acenaphthene similarly yields β -5-acenaphthoyleacrylic acid, m.p. 154—155°, and on cyclisation an acid, m.p. 216—217°. C. HOLLINS.



Manufacture of α -anthraquinonyl ketones. I. G. FARBENIND. A.-G. (B.P. 273,774, 5.7.27. Ger., 5.7.26. Addn. to B.P. 271,884; B., 1928, 665).—Benzanthrones containing alkyl groups in positions 2 and 3, or an alkyl or a nitrile group in position 2 or 3, are oxidised with chromic acid. 3-Methylbenzanthrone gives 1-anthraquinonyl methyl ketone, m.p. 160°; 2-methylbenzanthrone yields α -1-anthraquinonyl- β -methylglyoxal, m.p. 195°; both are obtainable from 2:3-dimethylbenzanthrone. Anthraquinone-1-carboxyl cyanide [1-anthraquinonylglyoxylonitrile], m.p. 297° (decomp.), is formed, together with a substance, m.p. 276—277°, from 3-cyanobenzanthrone. C. HOLLINS.

Manufacture of aminoanthraquinones, their homologues, and substitution products from the

corresponding leuco-amino-compounds. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 298,279, 11.7.27).—The leuco-derivatives of 1:4-diaminoanthraquinones are oxidised by heating in nitrobenzene at 140—180° in presence of a small quantity of a basic or acid catalyst, *e.g.*, thionyl chloride, piperidine, triethylamine, benzoyl chloride, etc. C. HOLLINS.

Manufacture of new intermediate compounds and of azo dyes therefrom. BRIT. DYE STUFFS CORP., LTD., and M. MENDOZA (B.P. 297,855, 27.5.27).—The processes of B.P. 245,865 and 262,243 (B., 1926, 233; 1927, 101) are extended to the manufacture of amino-sulphones from 5-substituted salicylic acids, including 2:3-hydroxynaphthoic acid, and to azo dyes therefrom. 3-Chlorosulphonyl-5-methylsalicylic acid, from *p*-cresotic acid, is reduced to 3-sulphino-5-methylsalicylic acid, m.p. 170°; 5-chloro-3-sulphinosalicylic acid, m.p. 200—201°, and 2-hydroxy-1-sulphino-3-naphthoic acid are similarly prepared. These are condensed with 4-chloro-3-nitrobenzenesulphonic acid, 2-chloro-5-nitrobenzenesulphonic acid, 1-chloro-2:4-dinitrobenzene, or 1-chloro-2:6-dinitrobenzene-4-sulphonic acid to give nitro- or dinitro-sulphones, which are reduced to amino- or diamino-sulphones; 2:4-dinitro-2'-hydroxy-3'-carboxy-5'-methylidiphenyl sulphone melts at 284—286°, the 5'-chloro-compound at 266—268°, and 2:4-dinitrophenyl 2-hydroxy-3-carboxy-1-naphthyl sulphone at 260° (decomp.). Azo dyes which show little change in shade on chroming are obtained by coupling the diazotised aminosulphones or monoacylated diaminosulphones with coupling components, or the diaminosulphones with diazo compounds, or tetrazotising the diaminosulphones derived from 1-chloro-2:6-dinitrobenzene-4-sulphonic acid and coupling with coupling components. The dyes are useful for chrome printing, and as chrome wool colours. C. HOLLINS.

IV.—DYE STUFFS.

Reactions of dyes with nitrous acid. J. V. DUBSKÝ and A. OKÁČ (Z. anal. Chem., 1928, 75, 92—111).—The characteristic colours produced by diazotising certain dyes and materials from which they are derived afford a delicate method for the detection of nitrous acid. The colour in most cases is intensified or modified by coupling the diazo compound with substances of a phenolic character or with acid dyes; this is especially marked with substances having a free *para*-position. The reaction of nitrous acid with over 100 compounds has been examined, and the further behaviour on coupling with numerous other compounds is recorded. The sensitivity of the test is of the order of 10^{-6} — 10^{-7} . A. R. POWELL.

See also A., Nov., 1171, Relationship between absorption spectrum and chemical constitution of azo dyes (BRODE). 1187, Viscosity of some dyes (BANERJI and DHAR). 1237, Diphenylamine derivatives (ORLOV). 1238, Azo dyes from acetoacetanilides (FIERZ-DAVID and ZIEGLER). 1250 and 1251, Colouring matters of fungi (KÖGL and others). 1252, Colouring matter of paprika (ZECHMEISTER, VON CHOLNOKY, and VRABÉLY). 1255, 6-Aldehydodoumarin and derived dyes (SEN and CHAKRAVARTI). Plant colouring matters (KARRER and WIDMER; KARRER and SCHWARZ).

1256, Anthocyanin pigments of "Morning Glory" (KATAOKA). 1260, Diphenylpyridylmethane dyes (PLAZÉK and SUCHARDA). 1289, Photo-oxidation with anthraquinone (PFEILSTICKER).

Pigments of cacao beans. FINCKE.—See XIX.

PATENTS.

Manufacture of azo dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 297,884, 1.7.27).—Dyes from diazotised 5-nitroanthranilic acid are treated with sodium hydroxide and dextrose to reduce the nitro- to an azoxy- or azo-group. The products are suitable for after-coppering. The coupling components in the examples are γ -acid (black), J-acid (violet; diazotised on the fibre and developed with β -naphthol, black-blue), phenyl-J-acid (clear blue), acetyl-J-acid (violet), and 1-(6-sulpho- β -naphthyl)-3-methyl-5-pyrazolone (brick-red). C. HOLLINS.

Dyes. L. B. HOLLIDAY & Co., LTD., and C. SHAW (B.P. 298,098, 28.5.27).—An aminoazo compound is condensed with a phthalic acid or anhydride to give a phthalamic acid. The products from phthalic acid and aminobenzencazodiphenylamine (orange on wool, silk, cotton, and acetate silk) or aminoazobenzene (yellow on wool and acetate silk), and from tetrachlorophthalic acid and *p*-nitrobenzeneazo- β -naphthylamine, are described. C. HOLLINS.

Manufacture of dyes containing chromium. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,722, 25.6.27).—The dye, 4-chloro-2-aminophenol-6-sulphonic acid \rightarrow 2 : 4-dihydroxyquinoline, is heated at 110–120° with chromium formate solution to give a complex chromium compound which dyes wool in level bordeaux-red shades fast to washing, milling, and light (cf. B.P. 28,716 of 1909; B., 1910, 1197). C. HOLLINS.

Manufacture of derivatives of the triarylmethane series. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 298,101, 29.6.27).—Spirit-soluble salts of triarylmethane basic dyes, suitable for printing, are obtained by adding a hydroxynaphthoic acid (2 : 3- or 2 : 6-hydroxynaphthoic or 2-naphthol-3 : 6-dicarboxylic acid) and alkali to a solution of the hydrochlorides (magenta, malachite green, etc.). C. HOLLINS.

Production of an anthraquinone-hydrazine dye. W. SMITH, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 298,248, 1.4.27).—3-Chloro-1-bromo-2-aminoanthraquinone (B.P. 264,916; B., 1927, 246) is heated with sodium acetate and copper acetate at 160–170° in a diluent (*o*-nitrotoluene). The resulting dichloroind-anthrone gives bright reddish-blue dyeings fast to chlorine. C. HOLLINS.

Treatment of cashew nut-shell oil and products [colours] obtained thereby. HARVEL CORP., Assees. of M. T. HARVEY (B.P. 272,509, 7.6.27. U.S., 8.6.26).—Colours and colour bases ranging from deep red to violet are prepared by the interaction of cashew nut-shell oil and an ammonium compound or formaldehyde. The colours may be used for oil printing, or for mixing directly with varnishes, stains, or cellulose acetate. They are substantially unaffected by hydrochloric acid or by alkalis, but may be thickened to a paste by means of

oxidising or reducing agents, or by formaldehyde. When added to cellulose acetate or resins they act as plasticising and waterproofing agents. E. HOLMES.

Azo dyes (B.P. 297,855).—See III.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Oxidation of cellulose on exposure to light. II. V. SCHARVIN and A. PAKSCHVER (Z. angew. Chem., 1928, 41, 1159–1161; cf. B., 1927, 837).—Oxycellulose and carbon dioxide are produced simultaneously but not proportionally from cellulose exposed to the action of light in sealed tubes containing an atmosphere of oxygen. Small amounts of water and aldehyde-like substances are also formed. Dilution of the oxygen with inert gases reduced the rate of oxidation. No oxidation took place in pure nitrous oxide, carbon monoxide, ammonia, or hydrogen. A. G. POLLARD.

Sulphite-cellulose cooking [with a mixture of old and new cooking acids]. E. HÄGGLUND (Papier-Fabr., 1928, 26, 657–661).—No economy is effected by the use of a mixture of waste sulphite cooking liquor with freshly prepared liquor rather than a freshly prepared acid alone. Comparative experiments with freshly prepared acid and with a mixture of one third waste liquor and two thirds fresh acid of the same concentrations of lime and sulphur dioxide show that for the latter the colour of the product is inferior, the yield is smaller for the same lignin content, the ash content is higher for equal times of cooking, a greater degree of acidity of the liquor is attained, and cooking proceeds at a slower rate. It is concluded that the danger of overcooking is greater with mixed than with fresh liquor. For the manufacture of pulp for bleaching and for the production of artificial silk, mixed liquors should be avoided, especially in the latter case, since with increasing acidity of the liquor the α -cellulose content of the product falls. Better results are obtained by thorough mixing of liquid and pulp than when no agitation occurs, and the importance of efficient circulation in the cooker is emphasised. B. P. RIDGE.

Cymene produced in the sulphite-cellulose process. O. ÄSCHAN (Finska Kemistsamfundets Medd., 1927, 36, 123–125; Chem. Zentr., 1928, i., 2175).—The *p*-cymene obtained in the sulphite-cellulose process may contain *m*-cymene derived from sylvestrene by way of Δ^3 - and Δ^4 -carene. A. A. ELDRIDGE.

Experiments in producing cellulose ethers and esters with special reference to cellulose nitroacetate. H. A. GARDNER and C. A. KNAUSS (Amer. Paint & Varnish Manufs'. Assoc. Circ. No. 338, Oct., 1928, 657–672).—A general discussion on various ethers and esters of cellulose as possible substitutes for nitrocellulose in lacquers. Details of laboratory preparation of such products are given, special attention being paid to nitroacetates prepared by acetylation of the nitrate. The relative durability of lacquers containing cellulose esters and ethers is indicated by the results of exposure tests. S. S. WOOLF.

See also A., Nov. 1187, Denitration in artificial silk manufacture (NÁDAI). 1219, Action of alkali and carbon disulphide on xylan (HEUSER and

SCHORSCH). 1225, Cellulose (HESS and TROGUS). 1226, New degradation of cellulose (PRINGSHEIM, KASTEN, and SCHAPIRO). Action of highly concentrated nitric acid on cellulose (ANDRESS). Cellulose formate (UEDA and KATO). Sulphuric esters from cellulose (GEBAUER-FÜLNEGG, STEVENS, and DINGLER). 1227, Lignin and cellulose (FREUDENBERG, HARDER, and MARKERT). Acetylation of beech wood (SUIDA and TITSCH). Methylation of beech wood (VON WACEK). 1228, Structure of chitin (MEYER and MARK). 1229, Structure of silk fibroin (MEYER and MARK).

Action of bromine on cellulose, lignin, etc. FUCHS.—See II. Colloresin *D*. PFEFFER and GMELIN.—See VI. Rice straw. IWATA.—See XIX.

PATENTS.

Treatment of flax and similar fibrous materials. VICKERS, LTD., and O. D. LUCAS (B.P. 298,178, 5.7.27).—Scutched flax is boiled with a 5% solution of caustic soda, washed with water, and treated with a cellulose solvent, *e.g.*, ammoniacal copper or zinc chloride, of such strength as to exert a selective action on the fibre without dissolving or completely gelatinising it. The material is then boiled with a 1% solution of caustic soda with the addition of a plasticiser, *e.g.*, sulphonated fatty acids etc., washed, immersed in acid for 15–20 min., again washed to remove the acid, and dried, yielding a soft and lustrous fibre. F. R. ENNOS.

Improvement of vegetable fibres. CHEM. FABR. FRANKFURT-WEST LANDAUER & Co., and L. GUTLOHN (G.P. 446,435, 29.11.24).—Agave and palm fibres etc. are strengthened by treatment with solutions of alkali, alkaline-earth, or magnesium salts capable of modifying their structure, or by the deposition on them of silica or barium sulphate, or by a combination of both processes, and their lustre is improved by subsequent treatment with soaps or with fat or mineral-oil emulsions.

L. A. COLES.

Manufacture of tracing cloth. M. RENKER (B.P. 297,993, 29.10.27).—Tracing cloth is treated with a solution of cellulose esters or ethers in a solvent which does not dissolve the fats or oils used in finishing the cloth, *e.g.*, glycols or their derivatives, and dried at 50–80°. If the cloth is given a protective coating of oil, varnish, or resin, solutions of cellulose compounds in other solvents may be used. F. R. ENNOS.

Direct transformation of cellulose into defibred alkali cellulose for the manufacture of viscose. L. C. P. JARDIN (B.P. 286,620, 5.3.28. Fr., 5.3.27).—Cellulose in any form is torn to fine pieces in a suitable apparatus maintained at constant temperature, being at the same time sprayed with sufficient caustic soda solution to convert it into alkali cellulose.

F. R. ENNOS.

Cellulose xanthate process. W. MENDEL, Assr. to S. A. NEIDICH (U.S.P. 1,681,900, 21.8.28. Appl., 1.5.26).—The cellulose crumbs are freed from water and occluded air by means of a partial vacuum before treatment with carbon disulphide, and after treatment carbon disulphide fumes are removed in the same manner.

C. HOLLINS.

Spinning of artificial filaments, especially arti-

ficial silk from cuprammonium solutions of cellulose. K. GRUNERT, Assee. of GRUNERT & GIANNETTI (B.P. 282,670, 16.12.27. Ger., 24.12.26).—The precipitant is aerated with air or an inert gas, the larger bubbles of which are allowed to escape before entry of the liquid into the spinning device. F. R. ENNOS.

Treatment of waste liquors and precipitates therefrom obtained in the manufacture of cuprammonium silk. J. P. BENBERG A.-G. (B.P. 291,380, 25.5.28. Ger., 31.5.27).—The waste liquor or precipitate is heated at 130–140° with an amount of sulphuric acid in excess of that required to convert the copper into sulphate and to carbonise organic matter. The mixture of crude copper sulphate and carbon is separated from the liquor, and the former is purified by dissolution in water, filtration, and recrystallisation. F. R. ENNOS.

Recovery of waste gases [sulphur dioxide] in production of sulphite pulp. G. A. RICHTER, Assr. to BROWN Co. (U.S.P. 1,683,628, 11.9.28. Appl., 19.3.25).—The calcium bisulphite liquor for the treatment of the wood receives its sulphur dioxide first from burner gas, and then from recovery gas, being brought up to strength by sulphur dioxide released from time to time from the digester. The recovery gas unabsorbed by the bisulphite liquor is treated with fresh water in a second tower; the washings are used as condenser water, and thus lose their sulphur dioxide, which is passed with the burner gas to the bisulphite tower. C. HOLLINS.

Treatment of sulphite-wood-pulp liquor. W. E. B. BAKER (U.S.P. 1,685,800, 2.10.28. Appl., 25.2.22).—The liquor is treated with a sufficient amount of an alkaline earth to give p_H 9 or above, and the resulting solid normal sulphite is treated with a bisulphite solution containing free sulphurous acid to give sulphite liquor for re-use. F. G. CLARKE.

Production of non-hygroscopic solid material from sulphite-cellulose waste material. I. G. FARBERIND. A.-G. (Austr. P. 106,713, 8.1.25).—The liquor after removal of calcium and iron compounds is treated with oxidising agents, *e.g.*, potassium dichromate or sodium nitrite and hydrochloric acid, or is condensed with aldehydes, carboxylic acids, or amines, and is then evaporated to dryness and the residue ground to a powder (cf. F.P. 592,119; B., 1926, 188).

L. A. COLES.

Recovery of volatile plasticisers from celluloid etc. H. P. BASSETT (U.S.P. 1,681,692, 21.8.28. Appl., 11.6.27).—Celluloid scrap is covered with dilute nitric acid and steam-distilled under 1.3–2.6 atm. pressure. Camphor is removed and recovered, leaving nitro-cellulose in usable condition. C. HOLLINS.

Digestion of raw materials for papermaking etc. S. MILNE (B.P. 297,713, 26.3.27).—In the digestion of, *e.g.*, grass, with a suitable reagent, *e.g.*, caustic soda, the reaction is controlled by means of an externally-generated, transient *E.M.F.* of relatively high frequency, *e.g.*, that derived from electromagnetic radiation broadcast from a wireless station. J. S. G. THOMAS.

Spinning bowls or boxes, particularly for artificial silk. N. V. NEDERLANDSCHE KUNSTZIJDEFABR. (B.P. 297,450, 3.5.28. Holl., 22.9.27).

Apparatus for spinning solutions of cellulose esters and ethers. RUTH-ALDO CO., INC., Assees. of R. A. J. THIENOS (B.P. 283,140, 4.1.28. Fr., 4.1.27).

Fertiliser (U.S.P. 1,684,712).—See XVI.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Production of printed effects with combinations of anthraquinone and indigoid vat dyes. R. HALLER (Textilber., 1928, 9, 924—926).—The well-known process for discharging indigo with Rongalite can be successfully adapted by the use of ferrous sulphate and stannous chloride for the production of very fast anthraquinone vat dye discharges. Thus cotton fabric may be dyed in a jig with an indigoid vat dye (e.g., Ciba Blue 2B), then printed with a discharge paste consisting of 600 g. of an anthraquinone vat dye (e.g., Cibane Blue GCDN), 500 g. of Rongalite CL, 8700 g. of a discharge paste A (suitable for white discharges), 500 g. of ferrous sulphate, 200 g. of stannous chloride, and 200 g. of glycerin, steamed in an air-free Mather-Platt, and then passed through a hot solution of caustic soda and washed. Discharge paste A consists of 12 kg. of British gum, 3.5 kg. of glucose, 13 kg. of water, 10 kg. of Rongalite CL, 3—6 kg. of zinc oxide, 5 kg. of Leucotrope W, 0.5 g. of rape oil, 0.5 kg. of turpentine, and 1.8 kg. of a 50% solution of gum. A. J. HALL.

“Colloresin D” and its application in [textile] printing. E. PFEFFER and GMELIN (Textilber., 1928, 9, 666—669).—“Colloresin D” (I. G. Farbenind.) is a cellulose ether having the form of cotton wool; it swells in water at 40—50° and then dissolves on cooling to form a viscous solution suitable for replacing the thickening pastes of starch and gum tragacanth commonly used in printing. It is useful as a thickening agent in printing with vat dyes. A. J. HALL.

Naphthol AS dyes on cellophane [viscose film]. G. SCHWEN (Textilber., 1928, 9, 673—674).—The changes which occur in the size of the pigment particles and their migration within cotton and artificial silk fibres dyed with insoluble azoic dyes as described by Haller and Ruperti (B., 1926, 316), and Kayser (B., 1926, 784) have been re-investigated using dyed cellophane instead of fibres. Cellophane dyed with Naphthol AS-SW (0.1 g. per litre) and Fast Red KB base is transparent and homogeneous, only a few dye particles being visible in the ultra-microscope; when dyeing is effected with a solution containing 2 g. per litre of Naphthol AS-SW, the dyed cellophane is opalescent, and a large number of dye particles are visible in an ordinary microscope. Both dyeings become bluer and opalescent in proportion to the amount of pigment present when boiled for 15 min. in water or particularly in a solution containing 0.2% of soap and 0.2% of sodium carbonate, these changes being due to an aggregation of the dye particles; no aggregation occurs when the dyed cellophane is subjected to dry heat, thereby indicating that swelling of the cellulose is necessary for aggregation. The original shade and transparency of cellophane dyed with certain dye combinations were restored by heating the dry dyed cellophane for a brief period at about 250°, and it is concluded that the aggregation of dye particles as produced by soaping and their dispersion

by subsequent dry heat is intimately related to the m.p. of the dye. A. J. HALL.

Yellow and orange chromate discharges on indigo. Brown chromate discharges on indigo. N. WOSNESSENSKY (Sealed Notes 1916—7, 20.7.05. Bull. Soc. Ind. Mulhouse, 1928, 94, 469—471, 471—472). Report by P. BINDER (*Ibid.*, 472—473).—The discharges are obtained by means of the nitrosoamines of dichloroaniline and *p*-nitro-*o*-anisidine. A yellow discharge is obtained by impregnating indigo-dyed cotton fabric with a solution of ammonium acetate, then printing with a paste containing nitrosamine J (from dichloroaniline), neutral sodium chromate, sodium aluminate, pyrazolone, caustic soda, and thickening agents, steaming for a short period, and then leading the fabric through the usual “cutting liquor” containing sulphuric and oxalic acids. Bright orange discharges are similarly obtained using a printing paste consisting of nitrosamine O (from chloroanisidine), sodium chromate, β -naphthol, and caustic soda.

Brown discharges are obtained by printing indigo-dyed fabric with a paste of nitroso- β -naphthol, sodium chromate, chromium nitroacetate, and thickening agents, then steaming for 2 min. and leading through the usual “cutting liquor.” The colour of the discharge may be varied by using salts of iron or cobalt instead of chromium. BINDER reports that tendering occurs in the production of brown discharges. A. J. HALL.

Waterproof dyeings. K. ODA (Mem. Coll. Eng. Kyushu, 1928, 5, 71—92).—Grey to brown and black waterproof dyeings may be obtained by mordanting silk, viscose silk, and cotton yarns with iron, aluminium, or chromium in the usual manner and then treating with a dilute aqueous-alcoholic colloidal solution of the alcohol-soluble constituents of Japan lac, stearic acid, or palmitic acid; if desirable, the yarns may be first dyed by means of substantive dyes, e.g., Rhodamine B. The dyed yarns have a decreased capacity for absorbing water, and the viscose yarns have a considerably increased wet and dry tensile strength. Similar waterproof dyeings may also be obtained on mordanted yarns by after-treatment with gelatin and formaldehyde. A. J. HALL.

Mercerisation of sulphite-cellulose. V. I. SHAR-KOV (J. Chem. Ind. Moscow, 1926, 3, 1148—1149).—The maximum swelling occurs with 14% sodium hydroxide, or 17% potassium hydroxide, solution. The addition of alcohol or glycerin lessens the swelling. In the absorption of alkalis by cellulose equilibrium is reached in 30 sec. provided the solution has access to each fibre.

CHEMICAL ABSTRACTS.

Ricinic acid. SUNDER.—See XII. **Osage-orange and fustic extracts.** DESMURS.—See XV.

PATENTS.

Printing on textile fabrics. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,959, 14.9.27).—Vegetable mucilages (e.g., from carob seeds or salep bulbs) which are coagulable by alkali are used as thickeners in printing pastes of vat or sulphide colours in absence of alkali. The prints are developed by padding with alkali and reducing agent, drying, and steaming.

C. HOLLINS.

Dyeing or printing with vat dyes on acetate silk or mixed fabrics containing acetate silk. DURAND & HUGUENIN SOC. ANON. (B.P. 274,094, 7.7.27. Ger., 10.7.26).—The material is padded or printed with a solution of a soluble leuco-ester of a vat dye, *e.g.*, an Indigosol, dried, and steamed. The leuco-ester in this manner penetrates and is fixed on the fibre, and may be developed by heating with acid and nitrite at 70–80°; the nitrite may be present in the padding or printing solution. C. HOLLINS.

Dyeing of cellulose esters and ethers. BRIT. DYESTUFFS CORP., LTD., L. G. LAURIE, F. W. LINCH, and E. H. RODD (B.P. 297,897, 5.7.27).—Acetate silk etc. is dyed in bright green shades, fast to light and to alkaline storage conditions, by application of dyes of the malachite- or brilliant-green type containing in the "aldehyde" nucleus an alkyl group in position 2 and alkyl or alkoxy groups in positions 4 and/or 5. Examples are the 2:4- and 2:5-dimethyl and 5-methoxy-2-methyl derivatives of malachite- or brilliant-green. C. HOLLINS.

Mordant dyeing of cellulose derivatives [esters and ethers]. BRIT. CELANESE, LTD., G. RIVAT, and E. CADGENE (B.P. 273,693, 22.6.27. U.S., 30.6.26).—Acetate silk etc. is mordanted in presence of swelling agents such as formic acid, acetic acid, furfuraldehyde, phenol, pyridine, especially a mixture of furfuraldehyde with 3–5 pts. of acetic acid. C. HOLLINS.

Peroxide solutions (B.P. 277,628).—See VII. **Colouring rubber** (B.P. 298,364).—See XIV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Efficiency of different contact substances for the sulphuric acid contact process. I. B. NEUMANN, H. PANZNER, and E. GOEBEL (Z. Elektrochem., 1928, 34, 696–704).—A detailed description is given of an apparatus used for comparing the yield of sulphuric acid obtained on passing a mixture of sulphur dioxide and air, containing 7% of the dioxide, at rates ranging from 150 to 750 c.c./min. and at temperatures up to 750°, over various catalysts, the catalyst carrier being either asbestos or pumice. The method adopted for keeping the composition of the sulphur dioxide-air mixture constant is outlined. The sulphuric anhydride is passed into water, the "fog" being absorbed in a tube packed with glass wool. Tests made with platinised asbestos (7% Pt) show that, with a streaming velocity of either 150 or 300 c.c./min., a maximum yield of 99.6% is obtained at 425°. With silver vanadate as catalyst, maximum yields of 97.0, 92.5, 86.0, and 81.0% are obtained at 450° (streaming velocity 150 c.c./min.), 465° (300 c.c./min.), 487.5° (500 c.c./min.), and 507° (750 c.c./min.), respectively. With vanadic acid practically no reaction takes place below 400°. Maximum yields of 90.0, 79.5, 71.3, and 63.6% are obtained at 512° (streaming velocity 150 c.c./min.), 550° (300 c.c./min.), 575° (500 c.c./min.), and 600° (750 c.c./min.), respectively. Results very little better than those given by vanadic acid are obtained by the use of vanadic acid containing copper vanadate

maximum yields of 91.3, 80.9, 70.5, and 68.2% being found at 512° (150 c.c./min.), 550° (300 c.c./min.), 550° (500 c.c./min.), and 575° (750 c.c./min.), respectively. With the object of explaining the great difference between the efficiencies of silver and copper vanadates, the catalytic action of silver was tested, but found to be very poor, probably owing to the formation of silver sulphate. Tungstic acid is a poorer catalyst than vanadic acid, giving very little yield below 600°. The mechanism of the vanadic acid catalysis is discussed.

L. L. BIRCUMSHAW.

Rapid determination of phosphate in technical citric and tartaric acid liquors. F. PERCIABOSCO (Atti II. Cong. Naz. Chim. Pura Appl., 1926, 1371–1373; Chem. Zentr., 1928, i, 2275).—The liquor (10 c.c., or 50 c.c. of purer solutions) is diluted to 300 (or 200) c.c.; a slight excess of ammonia, then 25 c.c. of ammonium citrate solution (unless citric acid liquor is being analysed) is added, and treated while hot with 25 c.c. of magnesia mixture.

A. A. ELDRIDGE.

Purification of hydrogen [for use in the manufacture of ammonia]. G. LEFORT DES YLOUSES (Sci. et Ind., 1927, 11, 53; Chem. Zentr., 1927, II, 2335).—Liquefiable impurities, *e.g.*, hydrogen sulphide and carbonyl sulphide, and the last traces of carbon monoxide are removed from hydrogen recovered from water-gas by treatment with liquid nitrogen under pressure at –180°.

L. A. COLES.

Utilisation of brass scrap in the manufacture of copper sulphate. A. V. VINOGRADOV and V. I. VINOGRADOVA (J. Chem. Ind. Moscow, 1926, 3, 1229–1230).—The brass scrap was calcined, dissolved in hydrochloric acid, and the copper removed from the solution with zinc, or brass, or by hydrogen under pressure as cuprous chloride; the latter on interaction with barium sulphide yields cuprous sulphide which is ignited at 200–300° and dissolved in dilute sulphuric acid.

CHEMICAL ABSTRACTS.

Separation of zinc formaldehyde-sulphoxylate from zinc formaldehyde-bisulphite. C. SUNDER and A. KEMPF (Sealed Note 2223, 20.2.13. Bull. Soc. Ind. Mulhouse, 1928, 94, 473–474). Report by R. FLATT (*Ibid.*, 474–476).—A method of separation is based on the fact that zinc formaldehyde-sulphoxylate, but not zinc formaldehyde-bisulphite, is precipitated quantitatively from its aqueous solutions when heated with zinc oxide. *E.g.*, 500 c.c. of a solution containing zinc formaldehyde-sulphoxylate and zinc formaldehyde-bisulphite equivalent to 167 g. and 195 g. of the corresponding sodium salts, respectively, yielded after heating for 1 hr. at 45–50° 300 g. of a paste of the basic zinc salt equivalent to 160 g. of sodium formaldehyde-sulphoxylate, and the solution contained zinc salts equivalent to 180 g. of sodium formaldehyde-bisulphite and not more than 2 g. of sodium formaldehyde-sulphoxylate. FLATT confirmed substantially the efficiency of the separation, prepared in a pure form the basic zinc formaldehyde-sulphoxylate, and showed that when placed in water it hydrolyses in such a way that at equilibrium point the water in contact with it contains 0.14 g. of zinc formaldehyde-sulphoxylate per 100 c.c.

A. J. HALL.

Detection of heavy metals in magnesium salts. G. KOGAN (Pharm. Zentr., 1928, 69, 449—450).—The detection of small quantities of heavy metals by the formation of a brownish colour or precipitate on the addition of sodium or hydrogen sulphide to the acetic acid solution is vitiated if traces of iron in the ferrous state are present. In such cases the test should be made in a solution acidified with hydrochloric acid or a mixture of hydrochloric and acetic acids. F. R. ENNOS.

Formation of acetylene-yielding carbides at relatively low temperatures. F. FISCHER [with F. BANGERT] (Brennstoff-Chem., 1928, 9, 328—333).—Barium carbide has been produced by heating barium oxide, mixed with calcium oxide, in a current of methane, or by heating a mixture of barium oxide with carbon in a current of methane, or hydrogen, or *in vacuo*. The highest yields, over 60%, were obtained by heating either mixture in methane at about 1150° and under reduced pressure (12—18 mm.). Addition of a small quantity of ferric oxide in general affected the reaction favourably, but had no influence on the experiments carried out *in vacuo*. On treatment with water the product evolved not only acetylene but also hydrogen. It appears probable that the first product of the reduction is barium itself, which then is partly or wholly transformed into carbide or hydride according to the experimental conditions.

A. B. MANNING.

Ionic exchange in zeolitic silicates through the participation of hydrolytically dissociated salts. II. Natural silicates. H. KAPPEN and B. FISCHER (Z. Pflanz. Düng., 1928, A 12, 8—37; cf. Kappen and Rung, B., 1927, 364).—In order to investigate the possibility of the introduction of ions of hydrolysed salts into zeolites the following procedure was adopted. 10 g. of the finely-powdered mineral (chabasite, desmine, stilbite, natrolith, analcime, serpentine, apophyllite, wollastonite, and talcum) were treated with 200 c.c. of 0.5*N*-solutions of the following chlorides:—ferric, ferrous, aluminium, copper, and zinc. The solution was renewed after 24 hrs. After 10 days' action the minerals were washed with water till free from chlorides and air-dried. They were then treated with *N*-potassium chloride to determine exchange acidity. Similar treatments with acid and the effects of diminution of particle size and of time of reaction were also studied. Soil and trass (volcanic earth) were treated similarly. The aluminosilicates chabasite, desmine, and stilbite are especially reactive. Ferric iron, as on permutite, was almost without action on crystalline zeolites. The introduction of bases into crystalline zeolites was much more difficult than into permutite, trass, or soils. In contradistinction to soils and permutite, the quantity was increased by diminishing particle size (and hence surface area), by raising the temperature, and by raising the salt concentration. Exchange acidity was also displayed by these minerals after treatment with acids. In a detailed theoretical discussion it is maintained that the authors' views on the nature of soil acidity and the rôle of exchangeable aluminium are substantiated, although it is admitted that exchangeable hydrogen plays a part. Further work is needed to assign to each its measure of importance.

H. J. G. HINES.

Isotherms and isobars for air separation studies. B. F. DODGE (Chem. Met. Eng., 1928, 35, 622).—Tables are given showing the vapour composition and pressure at various constant temperatures as a function of the composition of a liquid oxygen-nitrogen solution. Also tabulated are vapour compositions and temperatures as a similar function at constant pressures. If y , x are the vapour and liquid compositions, then for each isobar $y(1-x)/x(1-y)$ is very nearly a constant and may be used for approximate calculations. C. IRWIN.

Determination of carbon dioxide [in air]. H. LUNDEGÅRDH (Z. Pflanz. Düng., 1928, A12, 1—4). P. HASSE (*Ibid.*, 4—7).—Polemical. H. J. G. HINES.

See also A., Nov., 1169, **Condition of radium salts after storage** (FRANCIS and PARSONS). 1176, **Tungsten carbides** (BECKER). 1183, **Adsorption and dissolution phenomena in precipitations** (BALCAR and STEGEMAN). 1186, **Alcosol of silicic acid** (INABA). 1190, **Thermal decomposition of Bayer's alumina** (PARRAVANO and MALQUORI). 1191, **System barium carbide-barium oxide-carbon-carbon monoxide** (THOMPSON). 1195, **Catalytic conversion of thiosulphates into polythionates** (KURTENACKER and CZERNOTZKY). 1196, **Thermal decomposition of ammonia** (BURK). **Decomposition of hydrogen peroxide at surfaces** (WRIGHT and RIDEAL; WRIGHT). 1197, **Decomposition of ammonia by high-speed electrons** (McLENNAN and GREENWOOD). 1199, **Crystalline aluminium hydroxide** (FRICKE). 1201, **Tungsten carbonyl** (JOB and ROUVILLOIS). 1204, **Determination of fluorine** (KANDILAROV). **Determination of sulphuric acid in presence of antimony** (VON FINÁLY). **Determination of polythionates** (RIESENFELD and SYDOW). **Detection of pentathionate in presence of sulphurous acid** (CZERNOTZKY). **Elimination and determination of nitrogen in argon** (LEU). **Determination of nitrogen** (SAIKO-PITTNER). **Determination of phosphoric acid** (SOMEYA). 1205, **Micro-determination of potassium** (LEULIER, VELLUZ, and GRIFFON). **Determination of potassium** (DALSGAARD). **Micro-determination of sodium** (DI BENEDETTO and MARENZI). **Electrometric determination of calcium** (CORTEN and ESTERMANN). 1206, **Volumetric determination of mercuric oxide** (RÂY and DAS-GUPTA). **Analysis of sodium and barium tungstates** (JANDER and MOJERT). **Determination of thorium** (HECHT). 1207, **Determination of antimony in presence of arsenic** (RATHSBURG). 1209, **Extraction of krypton and xenon from air and from gases dissolved in water** (CLAUDE). 1230, **Determination of cyanides and basic cyanides of mercury** (IONESCO-MATIU and CARALE). 1280, **Toxicity of lead compounds** (BISCHOFF and others).

Fire extinguishers. BRANDL.—See I. **Radiant energy from flames.** GARNER.—See II. **Dissociation of silicates.** GUILLIN.—See XVI.

PATENTS.

Continuous concentration of dilute hydrochloric acid. SOC. ANON. DES DISTILLERIES DES DEUX-SÈVRES (F.P. 625,511, 30.11.26. Belg., 1.12.25).—The acid is distilled in the presence of salts soluble in water, e.g.,

calcium, magnesium, or zinc chloride, or of aliphatic or aromatic sulphonic acids. L. A. COLES.

Production of esters of silicic acid. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 298,778, 24.10.27).—Silicon tetrachloride is treated with alcohols in the presence of non-reacting diluents, *e.g.*, benzene. L. A. COLES.

Apparatus for manufacture of sodium sulphate and hydrochloric acid. E. V. BENJAMIN, Assr. to MYLES SALT Co., LTD. (U.S.P. 1,681,760, 21.8.28. Appl., 2.1.25).—Sodium chloride is suspended in baskets in a series of decomposing chambers below ground level, where it is treated with a current of hot sulphur dioxide, steam and air entering at the top and leaving at the bottom of each chamber in such a way that fresh chloride is subjected to the action of the more exhausted gases. A continuous plant is described. C. HOLLINS.

Removing monoalkaline-earth phosphates from monoalkali phosphate solutions. T. BENCKISER, A. REIMANN, A. REIMANN, JUN. (J. A. BENCKISER), and F. DRAISBACH (B.P. 298,436, 21.2.28).—Monoalkaline-earth phosphates are removed from the solution of monoalkali phosphates obtained by the treatment of crude phosphates with sulphuric acid by the addition of dialkali phosphates. The latter are added either in theoretical quantity or in slight excess, and the solution is heated at 150–200° and under 5–15 atm.; the resulting solution is entirely free from calcium and contains only 0.4–0.7 pt. of MgO to 100 pts. of P₂O₅. P. E. L. FARINA.

Production of barium ortho- or tri-silicate. RHEINANIA-KUNHEIM VER. CHEM. FABR. A.-G., Assees. of F. ROTHE and H. BRENEK (G.P. 449,737, 28.8.26).—Barium carbonate is heated with silica or barium metasilicate in the presence of steam, which may be produced by the combustion of gases containing hydrogen for supplying the necessary heat. L. A. COLES.

Manufacture of calcium polysulphide. C. D. WOOD, Assr. to LOS ANGELES CHEM. Co. (U.S.P. 1,685,895, 2.10.28. Appl., 1.9.26).—Sulphur is ground in the presence of lime and hot water. F. G. CROSSE.

Treatment of fines of earthy materials. R. W. HYDE, Assr. to DWIGHT & LLOYD METALLURGICAL Co. (U.S.P. 1,684,958, 18.9.28. Appl., 30.4.25).—A mixture of an alkaline-earth carbonate in small particles with a small proportion of solid fuel is exposed in a uniform layer and calcined so that heated combustion gases from an independent source pass through the entire layer at a constant rate. The solid fuel is thereby burnt, and the carbonate, heated and maintained above its decomposition temperature but below its fusion point, is converted into oxide. W. G. CAREY.

Treatment of slag and the like for the production of aluminium sulphate and other by-products. M. ODLING and A. A. STREET (B.P. 298,141, 4.5.27).—Blast-furnace slags, natural rocks, or slags of similar nature are crushed, mixed with water, and heated in pans with sulphuric acid under conditions that render the silica insoluble; the mass is lixiviated with water, filtered or decanted from insoluble matter, and evaporated to recover the aluminium sulphate. Lixiviation

may be repeated and the resulting solutions used as desired. The conditions for treating the slag are determined by the nature of the latter. Generally 2½ gals. of water to 1 cwt. of slag and seven eighths of the theoretical sulphuric acid are used; the temperature required is 105°, and is obtained from the heat of reaction and from steam or waste gas. Suitable oxidising agents are added, if desired, to free the operation from noxious fumes. Instead of waxy hydrated aluminium sulphate, the crystalline basic sulphate may be produced by the addition of lime etc. after the reaction. The insoluble residue of calcium sulphate and silica may be either used for agricultural purposes or converted by heat into a hard wall plaster.

P. E. L. FARINA.
Separation of alumina from alum. G. S. TILLEY (U.S.P. 1,686,112, 2.10.28. Appl., 3.6.27).—Alum is heated at a temperature below which it would melt in its own water of crystallisation until more than one half of that water has been removed, after which the temperature is raised to drive off combined sulphur trioxide. H. ROYAL-DAWSON.

Removal of heavy metals other than iron from neutral or alkaline solutions of zinc compounds. SILESIA VER. CHEM. FABR., Assees. of H. KLEIN and W. FLEMMING (G.P. 450,285, 17.12.25).—The metals are precipitated by the addition of aqueous solutions of salts of a disubstituted dithiocarbamic acid.

L. A. COLES.
Separation of cobalt from [solutions containing] other materials [e.g., nickel]. S. C. SMITH (B.P. 298,301, 25.7.27).—Cobalt is selectively precipitated from solutions of cobalt and nickel salts (particularly sulphates) by means of lead peroxide without the addition of manganese. The cobalt and nickel are completely separated. The operation is conducted at the b.p.; cobalt is recovered from the residue by leaching with sulphuric acid, and the lead sulphate produced is reconverted into the peroxide, the process thus being a cyclic one. By suitably adjusting conditions the separation may be carried out in two stages, a small amount of cobalt being left in solution at the end of the first operation. The process may be applied to the treatment of ores, mattes, etc. P. E. L. FARINA.

Production of lead tetraethyl. W. S. CALCOTT, A. E. PARMELEE, and F. R. LORRIMAN (B.P. 280,169, 26.7.27. U.S., 3.11.26).—Ethyl chloride reacts at 30–45° with a lead alloy containing about 9% Na and 1% K.

L. A. COLES.
Manufacture of catalytic gels. SILICA GEL CORP., Assees. of E. B. MILLER and G. C. CONNOLLY (B.P. 280,939, 17.11.27. U.S., 19.11.26).—The hydrogel (as defined) is impregnated with the catalyst in soluble form (*e.g.*, the salt of a metal) by immersion for a definite time in a solution of required concentration. It is then converted into a hard, highly porous gel by dehydration, which may be effected by a current of air at 75–120° and subsequent increase to 300–400°. The salt is decomposed by further heat-treatment. The temperature of decomposition should not exceed 550°; where a metal catalyst is required the decomposition is carried out in a suitable reducing atmosphere. The hydrogel may be

impregnated with a mixture of salts and mixed hydrogels employed. A hydrogel is defined as a product giving a gel that will adsorb a minimum of 10% of its dry weight when in equilibrium with water vapour at 30° and 22 mm. of mercury. P. E. L. FARINA.

Purifying hydrogen or gases containing hydrogen. GEWERKSCHAFT DER STEINKOHLENZECHTE MONT-CENIS (B.P. 276,668, 23.8.27. Ger., 26.8.26).—The gases are treated with alkaline reacting substances (preferably soda-lyc, 5–15%) to remove carbon dioxide, hydrogen sulphide, sulphur dioxide, etc., and are then passed at increased temperature (150–450°) and pressure (70–800 atm.) over a catalyst, whereby the carbon monoxide is decomposed into water and methane. By exactly repeating the process the catalyst in the second vessel has a life of 10–100 times longer than the usual. The catalyst may consist of finely-divided metals of the iron group, or metals or oxides of chromium, zinc, manganese, or vanadium, either alone or containing compounds of alkali, alkaline-earth, and certain earth metals; they are deposited as nitrates on the carrier substance where they are decomposed and reduced. The process, suitably modified, is utilised for the synthesis of ammonia.

P. E. L. FARINA.

Carrying out exothermic chemical reactions under pressure and at raised temperatures. SOC. CHIM. DE LA GRANDE PAROISSE, AZOTE ET PROD. CHIM. (F.P. 626,491, 31.3.26).—Mixed gases, *e.g.*, nitrogen and hydrogen for the synthesis of ammonia, circulate through an annular space between the outer walls of a pressure chamber and the inner tube containing the catalyst so that they are preheated by the heat of reaction generated in the catalyst compartment. L. A. COLES.

Means for testing for carbon monoxide. E. W. HULTMAN (U.S.P. 1,684,587, 18.9.28. Appl., 23.3.26).—Test papers soaked in a solution of palladous chloride containing a hygroscopic salt to keep them permanently moist are claimed. A. R. POWELL.

Manufacture of stable solutions of peroxides, persalts, and peracids. OESTERR. CHEM. WERKE GES.M.B.H. (B.P. 277,628, 13.7.27. Austr., 16.9.26).—Solutions containing, or capable of giving, hydrogen peroxide are stabilised by the addition of an alkali pyrophosphate in the presence of an activator such as an alkali chloride and/or silicate. The specific actions of the stabiliser and activator are mutually dependent and intensified, the total amount added being about 4%. The acidity of the solution is restored after treatment by the addition of suitable acid. 30% hydrogen peroxide solutions thus treated may be kept at 70° for several hours, the extent of the decomposition being nil to 2–3%, according to the nature of the stabilising mixture employed. Such solutions, suitably diluted (0.5–3% H_2O_2), are of value in the bleaching industry in checking the action of catalysts which normally cause high peroxide consumption. Examples are given of the bleaching of straw, wool, and felt where a peroxide consumption of up to 100% is reduced to 10–20%. A particularly stable solution is obtained by adding Turkey-red oil to the other stabilising and activating constituents. P. E. L. FARINA.

Manufacture of hydrogen peroxide by cathodic reduction of oxygen. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 297,880, 30.6.27).—The electrolyte is saturated with oxygen under pressure and electrolysed with the pressure released, thereby obviating risks of explosion. By a continuous cycle of operations through the saturation vessel and electrolytic cell, a 3% solution of hydrogen peroxide is obtained with an average current efficiency of 80%. The process enables a greater current density to be employed, as this depends on the degree of supersaturation. P. E. L. FARINA.

Finely-divided tellurium. C. LEVADITT (F.P. 626,506, 2.4.26).—A 30% aqueous solution of dextrose is added to a 5% aqueous solution of sodium tellurite, and the mixture heated for 30 min. to boiling.

J. S. G. THOMAS.

Causticising unit. W. D. MOUNT (U.S.P. 1,685,929, 2.10.28. Appl., 16.10.22).—See B.P. 265,669; B. 1927, 409.

Heat exchanger (B.P. 297,509).—See I. **Manganese salts from gas purification** (G.P. 450,230).—See II. **Catalysts for synthetic methyl alcohol** (U.S.P. 1,681,750–3; B.P. 271,840 and 279,378). **Catalytic oxidation** (B.P. 298,142).—See III. **Waste liquors etc. from cuprammonium silk** (B.P. 291,380).—See V. **Blanc fixe** (G.P. 450,180). **White lead** (B.P. 298,520).—See XIII. **Salts from sea water** (U.S.P. 1,684,935).—See XXIII.

VIII.—GLASS; CERAMICS.

Beryllium glass. C. F. LAI and A. SILVERMAN (J. Amer. Ceram. Soc., 1928, 11, 535–541).—In order to determine the general melting and cooling effects of beryllium oxide on glass, a soda-beryllia-silica triaxial diagram was prepared. The batch was melted in a covered crucible in a gas-fired furnace for 9 hrs. at 1400–1430° and the glasses were carefully cooled in the furnace. The products were divided into five categories, *viz.*, (1) unmelted, (2) devitrified, (3) incipiently devitrified, (4) uniformly opaque, (5) transparent. From the clear glass area, four series of glasses were prepared for test purposes, especial precautions being taken with the heating and the cooling. The total time required for annealing was 18 hrs. Beryllium glass melts at a somewhat higher temperature than ordinary glass; it melts between 1400° and 1430° if the molecular ratio of its constituents Na_2O , BeO , and SiO_2 is limited between 1:1:4.25 and 1:1:4, respectively. Its annealing properties introduce no radical difficulties in manufacture. Its density is lower than that of corresponding calcium and magnesium glasses, but this value increases linearly with increasing beryllia content. The hardness of beryllium glass (6.2–6.7) increases only slightly with increasing beryllia content, greatly exceeds that of soda-lime glasses, and is greater than that of magnesium glass. The refractive index, which increases linearly with increasing beryllia content, is slightly higher than that of magnesium glass, but much lower than that of calcium glass. In the transmission of ultra-violet light, the value for beryllium glass is greater than that of ordinary glass, but less than that of magnesium glass. Ultra-violet light transmission de-

creases with increasing beryllia content. If hardness alone is required in a commercial glass, beryllium may be introduced as beryl, the presence of aluminium in which toughens the glass. A. T. GREEN.

Use of 8-hydroxyquinoline in silicate analysis.

J. ROBITSCHKE (J. Amer. Ceram. Soc., 1928, 11, 587—594).

—The methods of Berg (A., 1927, 639, 848) and of Hahn and Vieweg (*ibid.*, 639) have been applied to the determination of alumina, magnesia, and alkalis in silicates. The ground sample of silicate is fused in the normal way, and, after extracting the fused mass, the solution is neutralised, and slightly acidified with acetic acid. After the addition of ammonium acetate the aluminium is precipitated with a reagent prepared by grinding 8-hydroxyquinoline in acetic acid, diluting, adding ammonia until cloudy, cooling, and filtering. This precipitate, $\text{Al}(\text{C}_9\text{H}_6\text{ON})_3$, is collected on a filter, washed, dissolved in hydrochloric acid, and titrated with standard potassium bromide-bromate solution. Alternatively, the precipitate may be dried and weighed. After removing the calcium as oxalate, the filtrate is treated with sodium tartrate. On adding the reagent, neutralising with dilute sodium hydroxide solution, and warming to 70°, magnesium is precipitated as $\text{Mg}(\text{C}_9\text{H}_6\text{ON})_2$. After washing, this precipitate may either be dried and weighed or dissolved and titrated as above. A method for the determination of alkalis, using hydrofluoric acid for the removal of silica and 8-hydroxyquinoline for the elimination of alumina and magnesia, is described.

A. T. GREEN.

Method of determining free aluminium oxide in silicate mixtures and its application to the study of clays.

L. A. SCHMELEV (Trans. Ceram. Res. Inst., Moscow, 1928, No. 14. 24 pp.).—A small quantity of the powdered silicate mixture is treated in a test-tube with a hot alcoholic solution of alizarin-red, the solution having been previously saturated at the b.p. with boric acid to prevent coloration of the alkalis and alkaline-carths. The contents of the test-tube are boiled and decanted, and the residue is washed with a hot, saturated alcoholic solution of boric acid. The extent to which the residue has become stained red indicates the amount of free alumina present. Hydrargillite and diasporite do not react with the alizarin-red solution, but they become strongly coloured after heating for 2–5 min. in the Bunsen flame. No liberation of alumina from the kaolinite molecule was detected by this method between the temperature limits of 550° and 1100°.

F. SALT.

Deformation of refractory materials under load at high temperatures.

E. KÖHLER (Trans. Ceram. Res. Inst., Moscow, 1928, No. 12. 119 pp.).—The various methods of determining the refractoriness-under-load of refractories, as described in the literature, are critically reviewed, and preference is given to the method which measures the degree of deformation under a given load under the influence of a gradually rising temperature. Tests were carried out with apparatus consisting essentially of an electric carbon-resistance furnace provided with a system of levers for applying the load. A pointer recorded the movements of the test piece, magnified ten times, on a suitable scale. A constant load of 1 kg./cm.² was applied, and

the temperature rose at the rate of 300° per hr. up to 950°, and thereafter at 240° per hr. Temperatures were measured with a thermo-element, protected by a special tube passing through the bottom of the furnace, the junction of the element being level with, and in close proximity to, the lower third of the test-piece. The test pieces were cylinders 50 mm. high and 35.7 mm. in diam. The results of tests made on Russian clays showed that the finer the grog, the higher was the temperature of initial softening; a 40% deformation, however, occurred at a lower temperature with the fine grog mixtures than with the coarser grog mixtures. Bodies containing mixed grog sizes showed greater resistance to deformation than bodies with one fraction, whether fine or coarse. With mixtures made from one clay, a close relationship was noted between the maximum strength (cold), the temperature at which a 4% deformation took place under load, and the firing shrinkage corresponding to the maximum cold strength. A low-fired grog produced better cohesion between the grog grains and the bonding clay. The ratio of grog to bond, within the limits of 35% to 55% of bond, had no effect on the strength under load. Kaolin-grog bodies were also prepared and fired to different temperatures. Best results in the under-load test were obtained with bricks fired at cone 28.

F. SALT.

See also A., Nov., 1182, Adsorption of gases by glass (ZEISE). 1205, Colorimetric determination of silica (KING and LUCAS). 1211, Changes in clays through weathering (OKAZAWA).

Pulverising hard substances. VON WARTENBERG and others.—See I.

PATENTS.

Manufacture of ceramic products with pure white shades. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 297,939, 29.8.27).—The shaped ceramic materials are raised to a red heat or an elevated temperature in a stream of chlorine or in a gas containing chlorine.

H. ROYAL-DAWSON.

Production of refractory materials. A. WAGEMANN (B.P. 286,677, 8.3.28. Ger., 8.3.27).—The addition of artificial silicates (slag from metal smelting and dust-destroyer clinker) to the mix for siliceous and silica materials gives after burning a product with increased strength and resistance to slag attack. Up to 10% of such silicates is added according to the particular refractory. By these additions the amount of clay bond in rammed materials may be greatly reduced, giving a product of equal strength and density but with diminished shrinkage.

J. A. SUGDEN.

Manufacture of zircon refractories. LE R. H. MINTON (U.S.P. 1,684,739, 18.9.28. Appl., 27.6.25).—Zircon is ground wet in the presence of a deflocculant, such as tannic acid, lignin, or gallic acid, and coarse zircon is then added. A plastic mass is formed by means of dextrin, tar, gums, or similar binders, and the mixture is moulded and fired.

F. G. CLARKE.

Reinforced [laminated] glass. N. B. MATTINGLEY and A. REA (B.P. 298,423, 27.1.28).

Stabilisation of disperse systems (Swiss P. 121,097).—See I. Silicon carbide (U.S.P. 1,684,611).—See XI.

IX.—BUILDING MATERIALS.

Cottrell-Moeller process for dusts. VER EECKE.
—See I.

PATENTS.

Apparatus for calcining cement or lime. E. MEIER (G.P. 449,789, 31.12.24. Addn. to G.P. 445,554; B., 1928, 194).—The calcining zone in the apparatus described previously is provided with baffles for deflecting the course of the falling particles, the baffles being constructed of hollow fireclay bricks of square cross-section arranged with one of the edges pointing upwards. A current of cooling air is passed through the hollow cavities in the bricks. L. A. COLES.

Treatment of gypsum. RHENANIA-KUNHEIM VER. CHEM. FABR. A.-G., Assees. of F. ROTHE and H. BRENEK (G.P. 450,172, 3.8.26. Addn. to G.P. 438,172).—Alkali aluminium silicates are used as silica carriers in the prior process. Soluble alkali aluminium compounds are washed out of the sintered material after removal of sulphuric acid but before working up to cement. The proportions of the materials are so adjusted that they contain 2 mols. of CaSO_4 per mol. of SiO_2 , and alumina is removed almost quantitatively. L. A. COLES.

[Production of] cementitious material. E. R. HARRAP, J. A. CANN, and BELL'S UNITED ASBESTOS CO., LTD. (B.P. 298,270, 7.7.27).—The material comprises metal oxides (magnesium oxide) and soluble metal sulphates capable of forming oxysulphates or basic sulphates (ferrous sulphate), with or without the addition of retarding agents, e.g., boric acid. L. A. COLES.

Manufacture of materials for use in making or repairing roads and other surfaces. F. MORTON (B.P. 298,812, 20.12.27).—A mixture of pitch with 5–20% of pure bitumen is used as binding agent. L. A. COLES.

Stabiliser and plaster composition containing the same. A. H. GALLAGHER (U.S.P. 1,683,539, 4.9.28. Appl., 19.9.27).—A plaster having uniform setting properties is obtained by adding an alkali-hydrolysed, nitrogenous, organic material, such as glue or waste organic matter, together with re-ground set plaster, or other crystal-forming calcium sulphate. The former prevents too rapid setting, and the latter counteracts the effect of retarding substances or conditions. F. G. CLARKE.

Production of [heat-]insulating sheets. J. WEISS (B.P. 274,471, 13.7.27. Switz., 4.10.26).—Fibrous vegetable material, e.g., wood wool, is dipped in a mixture of kieserite solution and magnesium oxide and, after draining, is dried in moulds having perforated bottoms and covers. L. A. COLES.

Manufacture of heat- and sound-insulating material. P. H. USSING (B.P. 289,830, 12.4.28. Denm., 3.5.27).—Straw etc., after softening with alkalis and treatment with solutions of soluble soaps and aluminium salts to precipitate aluminium soaps on the fibre, is formed into sheets on wire screens and these are pressed and dried. L. A. COLES.

Siliceous composition flooring material. D. and R. F. MACDONALD (B.P. 298,290, 15.7.27).—Mixtures of silica with, e.g., iron, aluminium, calcium, and

magnesium oxides are heated to fusion and the product is ground to a suitable size. Additional colour may be added by coating the surface of the material with potassium or sodium silicate and pigments and again firing to form a coloured glaze. L. A. COLES.

Treatment of wood prior to impregnation. COMP. FRANÇ. DES ÉTABL. GAILLARD (F.P. 601,663, 4.8.25).—The wood is heated above 100° by means of hot air to expel volatile constituents and to open up the pores, and the sap is then expelled by treating the wood with hot water, creosote oil, or sodium fluoride or copper sulphate solution under pressure followed by treatment *in vacuo* to withdraw the liquid from the pores. L. A. COLES.

Preservation of wood. Z. HADNAGY and L. LANYI (F.P. 624,258, 1.3.26).—Railway sleepers etc., after impregnation with preservatives if desired, are coated with cement resistant to water and salt solutions. L. A. COLES.

Antiseptic material for the preservation of wood. G. FRANCIOSI (F.P. 608,576, 28.12.25. Ger., 29.12.24).—The material comprises a solution of peat tar in not less than its own weight of tar oil. Up to one third of the peat tar may be replaced by wood tar, and zinc chloride may also be added. L. A. COLES.

Mixtures of pulverulent materials (B.P. 273,768).
—See I. Naphthenic compound (U.S.P. 1,681,657).
—See III. Glueing wood (B.P. 279,087).—See XVII.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Siemens-Martin open-hearth furnace with mixed-gas heating. W. HEILIGENSTAEDT (Stahl u. Eisen, 1928, 48, 1465–1472).—The firing of open-hearth steel furnaces with producer gas is compared with the procedure necessary with mixed gas (coke-oven gas and blast-furnace gas). In the latter case the gas must be preheated to 1000 – 1100° to decompose methane, whereas producer gas need be preheated only to 450° . Hence with mixed gas a much more efficient heat-interchange system is required. The heat balance in both cases is discussed at some length, together with the necessary changes in the design of the furnace and regenerator. A. R. POWELL.

Heat content and sp. heat of some [blast-furnace and open-hearth] slags at high temperatures. S. UMINO (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 985–996).—The heat content and sp. heat of three blast-furnace slags and of three open-hearth furnace slags have been determined up to 1500° . The mean heat content at 1500° is about 400 g.-cal./g., and the mean sp. heat increases from about 0.18 at 100° to 0.28 at 1550° . A. R. POWELL.

Energy losses of a 7-ton and of a 10-ton Héroult furnace. N. WARK; also H. KLINAR, O. REINHOLD, and N. WARK (Arch. Eisenhüttenw., 1928–9, 2, 145–150, 151–153; Stahl u. Eisen, 1928, 48, 1441–1442).—The heat balance of two Héroult steel furnaces has been determined; for the 7-ton furnace 20.8% of the heat input is lost by radiation, 14.7% in the cooling water, and 11.6% in the conductors. A. R. POWELL.

Energising action of carbonates contained in carburising mixtures. G. TAKAHASHI (Sci. Rep. Tôhoku Imp. Univ., 1928, 17, 883—926).—The rate of carburisation of iron by carbon and by carbon monoxide in the presence of carbonates of the alkalis and alkaline-earth has been investigated. That the accelerating effect of carbonates on cementation is not due to reduction by carbon followed by interaction between carbon monoxide and the iron is shown by the fact that carburisation is more energetic with carbonate-carbon mixtures than in a rapid current of pure carbon monoxide. Again, carburisation is accelerated by carbonates even at temperatures below the dissociation temperatures of the carbonate used, and when carbon monoxide is used as the carburising agent in place of carbon. In the latter case, when no iron is present, decomposition of carbon monoxide into carbon dioxide and finely-divided carbon takes place at carburising temperatures, and it is this active carbon which diffuses into iron during cementation at such increased velocity. Hence the rapid action of the carbon-carbonate mixture is due to the progressive formation from the carbon of carbon monoxide by the action of small quantities of oxygen or carbon dioxide and its almost immediate decomposition by the carbonate into an active form of carbon having a high rate of diffusion in contact with ferrous metals. A. R. POWELL.

Desulphurising action of manganese. Z. SHIBATA (Tech. Rep. Tôhoku Imp. Univ., 1928, 7, 299—303).—The degrees of desulphurisation of iron by manganese are calculated by means of the Nernst heat theorem for 1500°, 1600°, and 1700°, for various concentrations of manganese. C. W. GIBBY.

Mechanical properties of low-manganese steel. M. HAMASUMI (Tech. Rep. Tôhoku Imp. Univ., 1928, 7, 305—341).—Static tensile tests and notched-bar impact tests have been made on 21 low-manganese steels varying in composition from 0.1—0.4% C and 0.4—5.0% Mn. Cold-brittleness and blue-shortness are explained by the relative change of the maximum normal resistance and the maximum slip resistance of the crystals with respect to temperature. C. W. GIBBY.

Age-hardening of alloys and new age-hardening phenomena in iron. G. MASING (Arch. Eisenhüttenw., 1928—9, 2, 185—196; Stahl u. Eisen, 1928, 48, 1472—1473).—The phenomenon of age-hardening in alloys is discussed with especial reference to duralumin and beryllium-copper alloys (cf. Masing; also Dahl, B., 1928, 160). As the solubility of carbon in iron decreases from 0.03 to 0.04% at 700° to less than 0.01% at 20°, soft iron undergoes age-hardening when quenched from 650—700° and kept at the ordinary temperature for some days. This treatment increases the hardness from 120 to 185 Brinell and the tensile strength from 40.3 to 57.5 kg./mm.², whilst the elongation is reduced from 27.5% to 14%. A. R. POWELL.

Plastic deformation of metals. F. KÖRBER (Stahl u. Eisen, 1928, 48, 1433—1441).—A review of recent progress in the study of the mechanism of plastic deformation showing, with reference to numerous examples, the value of flow lines and recrystallisation

phenomena in interpreting the behaviour of mild steels during forging and rolling. The theories of plastic flow derived from the above-named considerations are shown to be confirmed by X-ray examination of plastically deformed metal. A. R. POWELL.

Ludwig-Soret effect in metallic alloys. II. M. BALLAY (Rev. Mét., 1928, 25, 509—520; cf. B., 1928, 787).—It is shown thermodynamically that the Ludwig-Soret effect should depend on the heat of dilution of the solution. The ratio c_1/c_2 of the concentrations in the colder and hotter parts of the solution should approach unity at infinite dilution, and not the ratio of the absolute temperatures. This is shown to be in much better agreement than the theory of van't Hoff with the experimental data recorded for potassium chloride, sodium chloride, and hydrochloric acid. C. W. GIBBY.

Separation and concentration of minerals by flotation. C. BERTHELOT (Rev. Mét., 1928, 25, 372—396, 411—426, 496—508).—A review of the theory and technique of flotation methods, of the plant and media used, and of the ores to which they are applied. C. W. GIBBY.

Lime scale as a concentrate. R. E. HEAD (Min. and Met., 1928, 9, 455—457).—Lime scale which formed on the sides of the cleaner cells of a Callow flotation plant treating auriferous copper ore was found to contain 95 oz./ton of gold, 35 oz./ton of silver, and 2.5% of copper, all of which were present as fine particles of free metal enveloped in a carbonaceous matrix which collected in the interstices of the crystalline calcium carbonate scale. A. R. POWELL.

Electrodeposition of silver with special reference to the use of sodium cyanide. E. B. SANIGAR (Trans. Faraday Soc., 1928, 24, 45—55).—Good deposits of silver with a satin surface may be obtained with baths containing sodium cyanide instead of potassium cyanide; a suitable bath contains 35 g. of silver nitrate, 40.4 g. of sodium cyanide (i.e., twice the theoretical to form the double salt), and 80 g. of sodium carbonate per litre. The conductivity of this solution at 25° is 9.08×10^{-2} mho compared with 14.25×10^{-2} mho for the usual potassium cyanide bath. Increasing the carbonate content of the bath results in a softer deposit, as also does an increase in the free sodium cyanide up to twice the combined sodium cyanide; more free cyanide than this tends to harden the deposit and to change its character. High current densities favour the formation of soft deposits, but in order to obtain smooth surfaces agitation is necessary. In replenishing the free cyanide content of potassium cyanide baths, sodium cyanide may be used, provided that pure salts are used and that care is taken to exclude impurities from the plating vat; in the presence of impurities, addition of the sodium salt to potassium cyanide vats often results in the formation of brown anode slimes, although such slimes are not produced in vats containing only sodium cyanide. A. R. POWELL.

Ageing of thermocouple wires. H. LENT and F. KOFER (Arch. Eisenhüttenw., 1928—9, 2, 173—176; Stahl u. Eisen, 1928, 48, 1473—1474).—The alteration in the calibration of base-metal thermocouples after

prolonged use in coke-oven and blast-furnace gases at 800° has been determined. In furnaces fired with coke-oven gas the calibration of couples composed of chronin-V2A steel, chronin-constantan, and chronin-NCT3 (17.5% Ni, 37.6% Cr, 0.48% Mn, remainder iron) altered rapidly, the *E.M.F.* for any temperature increasing rapidly with the time of exposure to the gases. In furnaces fired with flue gas from blast furnaces, however, all the above-named couples retained their calibration over long periods. Chronin-nickel couples withstand both types of gas-firing up to 1000–1100° for 5–6 weeks, and chronin-cekas (59.7% Ni, 11.2% Cr, 2% Mn, remainder iron) couples are stable for longer periods at 800°.

A. R. POWELL.

See also A., Nov., 1174, Crystallographic orientation of electrodeposited copper and nickel (TAMMANN and STRAUMANIS). X-Ray analysis of copper-tin alloys (WESTGREN and PHRAGMÉN). 1175, X-Ray analysis of silver-cadmium alloys (ÅSTRAND and WESTGREN). Fibrous structure of electrodeposited metals (TSUBOI). Recrystallisation of aluminium (TANAKA). X-Ray analysis of copper-magnesium alloys (RUNGQVIST, ARNFELT, and WESTGREN). 1178, Structure of quenched carbon-steel (KURDUMOV and KAMINSKY). Electrical resistance of alloys under pressure (UFFORD). 1181, X-Ray analysis of thallium-antimony alloys (PERSSON and WESTGREN). Recrystallisation of cast steel (HEIKE and WESTERHOLT). 1190, Systems cadmium-antimony and cadmium-lead (ABEL, REDLICH, and ADLER). Constitution of iron-tungsten and iron-molybdenum alloys (ARNFELT). 1191, System lead-antimony-cadmium (ABEL, REDLICH, and ADLER). 1195, Oxidative catalytic action of iron (WARBURG). 1196, Nickel catalyst for continuous hydrogenation (SVIZUIN). 1201, Iron and tin (KNOWLTON). 1204, Determining chromium in chromium steels (SOMEYA). 1206, Determination and separation of chromium, iron, aluminium, and phosphorus (JÄRVINEN). 1207, Determination of vanadium in presence of chromium, tungsten, and iron (WILLARD and YOUNG).

Cottrell-Moeller process for dusts. VER ECKE.—See I. Brass scrap. VINOGRADOV and VINOGRADOVA.—See VII. Water in relation to corrosion. HAASE.—See XXIII.

PATENTS.

Rotary furnace. E. AMME, K. DIENST, and D. J. UHLE (U.S.P. 1,685,972, 2.10.28. Appl., 25.2.27. Ger., 31.1.27).—A rotatable cooling drum, comprising a number of concentric cylinders separated by annular chambers communicating respectively with the inner part of the drum and with each other at opposite ends, is removably mounted in axial alinement with a rotary drum which can be heated. Elevating ribs project from the drums into the annular chamber, and disintegrating means are provided within the cooling drum.

J. S. G. THOMAS.

Centrifugal liquid crucible. J. MAXIMOFF and M. S. DE COSTA (U.S.P. 1,684,800, 18.9.28. Appl., 23.10.26. Fr., 24.12.25).—An apparatus for forming crucibles from liquid metals comprises a centrifugal

vessel with a refractory lining to contain the molten metal and a vertical electrode embedded in the bottom of the lining.

A. R. POWELL.

Steel manufacture. J. R. C. MARSH, Assr. to F. N. BARD (U.S.P. 1,684,841, 18.9.28. Appl., 19.1.22).—Molten iron is added to finely-divided, loose, graphitic material in such a manner as to effect a thorough mixing and completely to carburise the metal.

A. R. POWELL.

Reducing the corrosive action of acids on iron and steel in pickling baths. GOODYEAR TIRE & RUBBER Co., Assees. of L. B. SEBRELL (B.P. 298,390, 10.11.27. U.S., 21.10.27).—The attack of pickling acid on metal surfaces is minimised, without affecting the dissolution of oxide, by addition of about 0.1% of one of the products obtained by interaction of formaldehyde and hydrogen sulphide, e.g., dithiolmethane, dithioldimethyl sulphide, bis(thiolmethylthiol)methane, $\text{CH}_2(\text{SH})\cdot\text{S}\cdot\text{CH}_2\cdot\text{S}\cdot\text{CH}_2\cdot\text{SH}$, and the cyclic sulphides derivable from these.

C. HOLLINS.

Proofing of iron and steel against rust. W. H. COLE (B.P. 298,328, 22.8.27).—The metals are treated, in an autoclave, either fixed or movable, with the mixed phosphate solution described in B.P. 289,906 (B., 1928, 488), at a pressure above atmospheric and at a temperature above the b.p. of the liquid under atmospheric pressure.

F. G. CROSSE.

Wet extraction of copper. F. DIETZSCH (B.P. 296,899, 26.9.27).—The ore is roasted if it contains sulphides and the product is leached with dilute hydrochloric acid or with a solution of sodium chloride saturated with sulphur dioxide. The leach liquor is freed from sulphur dioxide by passing it through a fresh quantity of ore and its copper content is separated by means of hydrogen sulphide generated by passing the roaster gases together with steam over red-hot coke. The liquor from which the copper has been removed is again saturated with sulphur dioxide and used for treating further quantities of ore.

A. R. POWELL.

Copper alloys. METALLBANK & METALLURGISCHE GES. A.-G. (B.P. 286,615, 5.3.28. Ger., 5.3.27).—Alloys exhibiting high mechanical strength, hardness, and toughness consist of 87–99% Cu, 0.5–10% Ni, and 0.5–3% of ferrosilicon.

F. G. CROSSE.

[Silver] alloys resistant to chemical action. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 297,665, 30.6.27. Addn. to B.P. 297,165; B., 1928, 863).—An alloy resistant to hydrochloric acid contains at least 78% of silver and up to 22% of thallium, or a mixture of thallium with one or more of the following: magnesium, zinc, arsenic, antimony, bismuth, tin, or silicon.

A. R. POWELL.

[Silver] alloys resistant to chemical action. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 298,122, 30.6.27. Addn. to B.P. 297,165; B., 1928, 863).—Alloys similar to those of B.P. 297,665 (preceding) consist of at least 78% of silver and up to 22% of a mixture of manganese with one or more of the elements thallium, magnesium, zinc, arsenic, antimony, bismuth, or tin, or of a mixture of manganese, thallium, and silicon with one or more of those elements. Alternatively,

the silver may be alloyed with up to about 22% of a mixture of manganese and silicon with one or more of the following: arsenic, antimony, bismuth, or magnesium. F. G. CROSSE.

Recovery of tin from cassiterite. E. A. ASHCROFT (B.P. 297,784, 24.6.27).—The crushed dried material is mixed with powdered iron, aluminium, magnesium, or with powdered iron-tin, nickel-tin, or nickel-iron-tin alloys, the quantity being sufficient or slightly in excess of that required for complete reduction; the charge is heated at 500–900° under non-oxidising conditions or in a wholly reducing atmosphere. Instead of iron, iron oxide mixed with sufficient carbonaceous fuel to reduce the oxide to metal *in situ* may be used at non-slugging temperature. M. E. NOTTAGE.

Magnetic alloys. W. E. BEATTY. From BELL TELEPHONE LABS., INC. (B.P. 297,938, 29.8.27).—Alloys of iron and cobalt containing 40–60% Co (preferably 50% Fe and 50% Co), heated at about 1100° for 2–3 hrs. and then cooled slowly in the furnace, have a permeability above that of Armco iron at a corresponding magnetising force. M. E. NOTTAGE.

Refining or purifying metals and alloys. F. W. CORSALLI (B.P. 297,759, 23.6.27).—The fluid metal or alloy is mixed with the necessary quantity of improving materials (*e.g.*, carbon, alloys, metals) or purifying and deoxidising substances (such as aluminium and soda) by allowing it to flow through a mixing device which, by itself, or in common with a collecting vessel into which it passes, is adapted to be set in vibration by swinging, rotating, or by impact alternately in opposite directions; or, alternatively, it may be fixed immovably. The mixing device is provided with main channels which are connected together by smaller inclined channels, and is rigidly connected with the collecting vessel, a common driving device being provided. The charge in the mixing and/or collecting devices is heated by means of an electric current, coal dust, oil, furnace or hot exhaust gases, or by the introduction of heat-generating materials such as "Thermit." The furnace gases are passed partly through an aperture and partly over the chamber or the collecting vessel into the mixing receptacle in a direction counter to that of the metal stream; or the hot exhaust gases are introduced and conducted through the inner chamber by means of ejector action. M. E. NOTTAGE.

Metallic composition and its manufacture. W. H. JUDY, Assr. to SUMET CORP. (U.S.P. 1,686,277, 2.10.28. Appl., 9.8.23).—In the purification of lead, hydrogen to the exclusion of oxygen is introduced into the molten metal. H. ROYAL-DAWSON.

Flotation treatment of ores, coal, etc. W. SCHÄFER, and ERZ- u. KOHLE-FLOTATION GES.M.B.H. (B.P. 289,848, 16.3.28. Ger., 4.5.27).—The addition of solutions of polythionates especially of multivalent metals to the ore pulp assists the flotation of lead, zinc, copper, and antimony minerals. Galena is floated with ferrous dithionate and a hardwood tar oil; addition of a little sodium silicate inhibits the simultaneous flotation of blende, which is then subsequently floated by treating the pulp with copper sulphate and a zinc polythionate

solution. Copper pyrites is floated with sodium tetrathionate, sodium carbonate, and acetone oil, whereas iron pyrites remains unaffected by this treatment. Stibnite may be separated from mispickel by flotation in a pulp containing ferrous and manganous dithionates together with a little copper sulphite and sulphuric acid.

A. R. POWELL.

Concentrating oxidised ores and minerals. F. A. BIRD (U.S.P. 1,686,064, 2.10.28. Appl., 30.8.26).—Ore, prior to flotation concentration, is ground in a mill free from metal surfaces which might make contact with the ore, and is then subjected to a sulphidising agent.

H. ROYAL-DAWSON.

Production of coated metal articles, *e.g.*, stereo-type plates. C. C. WALLER (U.S.P. 1,684,565, 18.9.28. Appl., 7.2.24).—A copper coating is produced upon a stereotype plate by impregnating a fibrous blank with an aqueous emulsion containing copper sulphate, hydrated aluminium silicate, and gum arabic, impressing the desired characters thereon at 115°, casting molten type metal against the matrix so formed, and removing the casting and cleaning its surface. A. R. POWELL.

Operation of cupolas. F. B. DEHN. From GRIFFIN WHEEL CO. (B.P. 297,715, 20.4.27).—See U.S.P. 1,627,536; B., 1927, 683.

Finely-divided powders (U.S.P. 1,685,956).—See I. Treatment of slag (B.P. 298,141).—See VII.

XI.—ELECTROTECHNICS.

See A., Nov., 1171, Photo-electric emission of water and aqueous solutions (COUNSON and MOLLE). 1178, Resistance of alloys under pressure (UFFORD). Change of resistance of lead by action of radium (PRASAD and BASU). Conductivity of vaporised potassium chloride (PECZALSKI and CHICHOCKI). 1181. Conductivity of tellurium (KRAUS and JOHNSON). 1184, Determination of electro-endosmosis (SCHÖNFELDT; ILLIG and SCHÖNFELDT). 1192, Conductivity determination (REDLICH). Light-sensitivity of selenium cells (BARNARD). 1192, *E.M.F.* between copper and its amalgam (OKU). Lead-mercurous iodide cell (VOSBURGH). 1197, Electrochemical reduction of solid electrodes (FISCHBECK and EINECKE).

Cottrell-Moeller process for dusts. VER EECKE.—See I. Electrodeposition of silver. SANIGAR.—See X. Antimony electrode and soils. SNYDER.—See XVI. Moisture testing. FISHER and JONES.—See XIX.

PATENTS.

Electric furnaces. A. D. KEENE, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,686,009 and 1,686,010, 2.10.28. Appl., [A] 24.9.26, [B] 14.1.27).—(A) Electric resistor heating units are supported and protected by a cast metallic frame held by and inter-fitting with upwardly extending, cast metallic frames having a number of openings. (B) A resistor support, comprising a block forming part of the lining of the furnace wall and having portions projecting from the face of the wall, is carried by the furnace wall. Elongated members fit in the block and extend outwards over the projecting portions. J. S. G. THOMAS.

Electric [induction crucible] furnace. C. B. FOLEY, Assr. to C. B. FOLEY, INC. (U.S.P. 1,685,914, 2.10.28. Appl., 13.3.20. Renewed 23.2.28).—A uniformly dense, highly compressed block composed of finely-divided refractory material is carved to form half the crucible, near the bottom of which a tube, having a constricted passage between the tube itself, the bottom, and sides of the crucible, is formed as an integral portion of the block. Two such parts are united to form a complete furnace. J. S. G. THOMAS.

Electric furnace for producing silicon carbide. H. E. WHITE, Assr. to FEDERAL ABRASIVES Co. (U.S.P. 1,684,611, 18.9.28. Appl., 4.9.25).—The concrete end walls of the furnace, the hearth of which rests upon concrete supports, are unconnected with the latter. F. G. CLARKE.

Electrical gas purifier. SIEMENS-SCHUCKERTWERKE GES.M.B.H., Assees. of R. RÜDENBERG (G.P. 448,034, 7.5.25. Addn. to G.P. 426,149).—The capacity of the condenser arranged in parallel with the electrodes is so proportioned, and the amplitude of the pulsating direct voltage so chosen, that even the smallest voltage at the electrodes exceeds the ionisation potential by at least 20%. J. S. G. THOMAS.

Precipitating electrode for electrical gas purifier. SIEMENS-SCHUCKERTWERKE GES.M.B.H., Assees. of R. HEINRICH (G.P. 447,912, 15.2.24. Addn. to G.P. 438,972).—Angular or V-shaped slots, running principally in the vertical and horizontal directions, are provided. J. S. G. THOMAS.

Arrangement of electrodes for electrical gas purifiers. METALLBANK & METALLURGISCHE GES. A.-G. (G.P. 450,391, 29.8.25).—Precipitating electrodes comprise a number of single horizontal or vertical channels with intervening open slits arranged in a plane, and discharge electrodes extend parallel to the channels in the direction of the slits. Thus the slits throughout their length are in the region of the discharge of electricity from the discharge electrodes, which lie at the centre of a circular arc which touches the flange of a channel and the anterior edge of the channel next in the direction of the gas stream. J. S. G. THOMAS.

Method of cleaning electrodes of electrical gas purifiers. SIEMENS-SCHUCKERTWERKE GES.M.B.H., Assees. of H. HÖFLER (G.P. 448,505, 24.8.24. Addn. to G.P. 373,773).—Electrodes are removed and allowed to slide or roll down an inclined plane, and strike an obstacle. Alternatively, pendular electrodes are disturbed from their position of rest and strike an obstacle, *e.g.*, another electrode. J. S. G. THOMAS.

Protection of electrical gas-purifying plants. SIEMENS-SCHUCKERTWERKE GES.M.B.H., Assees. of J. FISCHER and F. MÜLLER (G.P. 450,392, 10.10.25).—A device operated by electric waves generated by a spark occurring at a spark gap on the chamber is caused to reduce the electrode potentials, or to cut off the current, or to operate a signal or switch. The device is screened so that it cannot be operated by electromagnetic waves other than those originated by the spark discharge in the chamber, and is non-resonant to the principal wavelengths generated by the spark discharge. J. S. G. THOMAS.

Electrical gas purification. H. ROHMANN and ELEKTR. GASREINIGUNGS GES.M.B.H. (G.P. 448,691, 30.10.21).—Suspended particles, owing to their different velocities or charges, are separately precipitated. Gaseous ions are precipitated by an electric field of such strength and dimensions and so arranged that suspended particles are not precipitated by the field. J. S. G. THOMAS.

Rectifier electrode. E. W. ENGLE, Assr. to FANSTEEL PRODUCTS Co., INC. (U.S.P. 1,686,316, 2.10.28. Appl., 7.1.27).—In an electrolytic device are electrodes of rhodium and tantalum, respectively, immersed in an electrolyte of sulphuric acid containing a small amount of metal of the iron group. H. ROYAL-DAWSON.

Galvanic battery. L. A. LEVY, and ALMEIDA ACCUMULATORS, LTD. (B.P. 298,289, 14.7.27).—A positive electrode of activated carbon, or an electrode associated, *e.g.*, packed, with activated carbon, is immersed in an electrolyte composed of a solution of a mixture of halides, *e.g.*, zinc chloride and zinc bromide, which just shows a definitely acid reaction towards Congo-red. J. S. G. THOMAS.

Production of electrodes emitting electrons at a relatively low temperature. REILIBUD RESEARCH & DEVELOPMENT Co., INC. (G.P. 449,672, 14.9.24).—A wire of platinum-iridium or platinum-nickel alloy, heated by a suitable electric current, is coated with barium carbonate or strontium carbonate by repeatedly dipping it into a solution of the carbonate in distilled water; the coating is converted into oxide by heating the wire strongly in the presence of oxygen. J. S. G. THOMAS.

Manufacture of incandescence cathodes for electric discharge devices. N. V. PHILIPS' GLOEI-LAMPENFABR. (B.P. 285,434, 13.2.28. Fr., 16.2.27).—A metal core is immersed in a bath of molten barium hydroxide and then heated, thus producing an electrode coated with barium oxide; the product is then heated in an oxidising atmosphere (*e.g.*, in oxygen) at about 1000°. H. ROYAL-DAWSON.

Insulating materials [for conductors of submarine cables]. A. R. KEMP, and WESTERN ELECTRIC Co., INC. (B.P. 298,694, 18.7.27. Addn. to B.P. 246,663).—Rubber compounds of different characteristics are mixed. Thus a mixture containing approx. 5% of sulphur and 95% of raw rubber is heated to 200–280°, and the product compounded with another mixture resulting from a mixture of 20% of sulphur and 80% of rubber by wt. similarly treated. If desired, the heating may be carried out after the final mixing. J. S. G. THOMAS.

Ion-concentration control. I. B. SMITH and E. A. KEELER, Assrs. to LEEDS & NORTHRUP Co. (U.S.P. 1,684,645, 18.9.28. Appl., 3.1.22).—A *P.D.* is produced by the variation in ion concentration, and the change in *P.D.* per unit change in ion concentration is magnified by mixing with the solution containing the ions a predetermined quantity of a suitable material, so that the slope of the potential-ion concentration curve is increased. The magnified *P.D.* is utilised to control the ion concentration of the unmodified solution. F. G. CLARKE.

Apparatus for detecting the presence of inflammable gas. W. M. THORNTON (B.P. 298,767, 11.10.27).

—An instrument of the type of a miner's electric lamp comprises two filaments mounted together, one of which is sealed in air and is normally used for illumination, whilst the other is exposed to the atmosphere under test. Means are provided for adjusting a variable resistance contained in the lamp casing, by relative movement of two parts of the casing, so that the two filaments appear equally bright, the amount of such movement indicating the percentage of inflammable gas present.

J. S. G. THOMAS.

Electric incandescence [projection] lamp. N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 285,069, 12.12.27. Holl., 11.2.27).

Augmenting the output of [dry] electric cells. G. C. FURNESS (B.P. 274,907, 25.7.27. U.S., 23.7.26).

Photo-electric cells. DE FOREST PHONOFILMS, LTD., Assees. of L. DE FOREST (B.P. 284,342, 27.1.28. S. Afr., 4.4.27).

Papermaking (B.P. 297,713).—See V. **Hydrogen peroxide** (B.P. 297,880).—See VII. **Liquid crucible** (U.S.P. 1,684,800). **Magnetic alloys** (B.P. 297,938).—See X. **White lead** (B.P. 298,520).—See XIII. **Irradiated milk** (B.P. 298,585).—See XIX.

XII.—FATS; OILS; WAXES.

Glycerides of fats and oils. XII. **Glycerides of lauric and myristic acids.** A. BÖMER and K. EBACH (Z. Unters. Lebensm., 1928, 55, 501—528. Cf. Bömer and Schneider, B., 1924, 478).—It was not found possible to isolate pure glycerides from palm-kernel oil by distillation in the vacuum of the cathode light (cf. Bömer and Baumann, Z. Unters. Lebensm., 1920, 40, 151) either by direct distillation of the oil after removal of free acids or by fractional crystallisation of the deacidified oil into 4 fractions and distillation of these separately. Addition of an equal weight of hardened sesamé oil or coconut oil produced no better results. The volatile glycerides of coconut oil can be distilled without difficulty from a mixture of the fat with ordinary or hardened sesamé oil. Trilaurin (m.p. 45·6°, corr.) and trimyristin (m.p. 56·2°, corr.) were isolated from laurel oil and nutmeg oil, respectively, by treatment of the oils with alcohol and repeated fractional crystallisation from acetone and ether. Laurel oil contains about 30% of trilaurin. No less soluble glyceride such as myristin was found. Nutmeg oil contains about 40% of trimyristin and 6% of free stearic acid. α -Lauro- $\alpha\beta$ -dimyristin (m.p. 43·3°) and α -myristo- $\alpha\beta$ -dilaurin (m.p. 38·0°) were prepared synthetically by the method of Fischer, Bergmann, and Bärwind (B., 1920, 790 A).

W. J. BOYD.

Lauric acid content of coconut and palm-kernel oils and the detection of these in mixtures of edible fats. J. GROSSFELD (Z. Unters. Lebensm., 1928, 55, 529—553).—Separation of lauric acid from other fatty acids by treatment with dilute alcohol or by means of the greater solubility of its lithium salt is unsatisfactory. The approximate lauric acid value (c.c. of 0·1N-acid per 5 g. of fat) of mixtures containing coconut oil, palm-kernel oil, and butter fat can be calculated from the formula $L = 3·3(V - B - 1·2C - V_K)$ where L is the lauric acid value, V the saponif. value, B the

butyric acid value, C the octoic acid value, and V_K the saponif. value of the pure fat in the absence of coconut oil, palm-kernel oil, and butter fat. V_K can, in general, be taken as 197 for edible fats. The lauric acid value so obtained was found to vary from 111 to 138 with a mean value of 126 for 22 samples of coconut and palm-kernel oils. In the case of butter fat it varied from 5 to 18 with a mean value of 11 for 10 samples. The content of coconut and palm-kernel oils in an edible fat mixture can be calculated from the lauric acid value according to the equation: coconut oil + palm-kernel oil = $0·79 \times (L - 0·6B)$.

W. J. BOYD.

Determination of iodine values by Margosches' method. G. BRACHMANN and A. MOROSOV (Oil Fat Ind. Russia, 1926, No. 2—3, 73—77).—Ethyl alcohol containing 1—2% of amyl alcohol was used as solvent, since it dissolves hard fats. CHEMICAL ABSTRACTS.

Validity of the Hagen-Poiseuille law in the pressure-viscosimetry of vegetable oils. P. SLANSKY and L. KÖHLER (Kolloid-Z., 1928, 46, 128—136).—The construction of a capillary viscosimeter is described in which the hydrostatic pressure is readily adjustable, and which is specially useful for investigating the pressure-viscosimetry of viscous liquids at slow rates of flow. Measurements with this viscosimeter and with a modified Ostwald-Krutz capillary viscosimeter showed only relatively small deviations from the Hagen-Poiseuille law in the region of small velocities of flow with all the vegetable oils examined, whether in the dried or undried state. Castor oil, in spite of its high viscosity, forms no exception in the course of its pressure-viscosity curve. In regard to the alteration in the properties of vegetable oils on drying, it is pointed out that the pressure-viscosity curve is not affected by the process of drying, and that the amount of the disperse phase in the dried oil must, therefore, be small.

E. S. HEDGES.

Validity of the Hagen-Poiseuille law for vegetable oils. W. OSTWALD, V. TRAKAS, and R. KÖHLER (Kolloid-Z., 1928, 46, 136—144).—Using the Ostwald-Auerbach overflow viscosimeter, a study has been made of the applicability of the Hagen-Poiseuille law to the viscosimetry of vegetable oils, mineral oils, and Voltol oils. Pure mineral oils showed no structure viscosity. Plant oils show little or no structure viscosity at the ordinary temperature, but show the phenomenon well at 5—7°. Partly polymerised plant oils and the Voltol oils exhibit marked structure turbulence at the ordinary temperature. It appears that structure viscosity is observed at a higher velocity of flow, the greater is the diameter of the capillary of the viscosimeter used.

E. S. HEDGES.

Action of carbon monoxide on the nickel catalyst in hydrogenation of fats. E. MASHKILLEISON (Oil Fat Ind. Russia, 1926, No. 2—3, 47—50).—In the presence of carbon monoxide the hydrogenation of oleic acid is retarded. CHEMICAL ABSTRACTS.

Hydrogenation of vegetable oils with water-gas. K. BUIOVSKI (Oil Fat Ind. Russia, 1926, No. 2—3, 45—47).—The water-gas was treated with 2N-sodium hydroxide solution, ferric oxide with 10% of sawdust, and hydrated lime with 10% of sawdust; the best

temperature was 220–250°. The iodine value of the oil was reduced from 78.3 to 66.9 after 5 hrs.; with hydrogen for 2.25 hrs. it was reduced to 35.7. A nickel catalyst was used. CHEMICAL ABSTRACTS.

Fatty acids of olive oil. K. II. BAUER and J. MITSOTAKIS (Chem. Umschau, 1928, 35, 275–277).—Lignoceric acid was identified in the "solid" fatty acids (separated by fractional precipitation of the lithium salts) from a carbon disulphide-extracted olive oil, but could not be traced in the acids from an expressed oil examined by the authors. E. LEWKOWITSCH.

Ricinic acid. C. SUNDER (Bull. Soc. Ind. Mulhouse, 1928, 94, 477–481).—Acid products obtained by treating castor oil with dilute caustic soda or acids remained liquid at the lowest temperatures obtainable by means of ice and salt, but a ricinic acid which solidified at low temperatures was obtained by effecting the saponification with caustic soda of *d* 1.334 at 100–120°; this latter acid is not identical with one previously described by Juillard (cf. B., 1893, 528). These new ricinic acids are capable of acting as mordants for alizarin dyes in the presence of aluminium salts. A. J. HALL.

Oil from mustard seed and husks. V. MOLODOVSKI (Oil Fat Ind. Russia, 1928, No. 2, 37).—The original oil, and the oil blown at 90–110° for 10, 40, and 75 hrs., respectively, have *d*₄¹⁵ 0.918, 0.924, 0.944, 0.966; Engler viscosity at 20° 9.9, 13.0, 42.0, 238.0; acid value 0.6, 0.36, 2.05, 4.0; saponif. value 188.4, 261.0, 244.0, 254.0; iodine value 116.2, 112.6, 85.3, 62.6; hydroxy-acids —, 2.92, 10.82, 24.04%; unsaponifiable matter 0.6, 1.57, 1.76, 4.78%. The husks contained moisture 5.15–8.2, protein 5.2–26.28, oil 11.8–14.1, cellulose 22.0, ash 5.58%.

CHEMICAL ABSTRACTS.

Elmseed oil. K. KARDASHEV (Oil Fat Ind. Russia, 1926, No. 2–3, 78–84).—The oil (20–25%) of the seeds of *Ulmus effusa*, Willd., and *U. scabra*, Mill., had Reichert–Meissl value 4.62–5.77, Polenske value 37.4–41.5, iodine value 15.9–25.0.

CHEMICAL ABSTRACTS.

Determination of the oil content of seeds. G. BRACHMANN (Oil Fat Ind. Russia, 1926, No. 9, 5–8).—The seed (4 g.) is kept in a stoppered bottle with ether (100 c.c. — expected vol. of oil); equilibrium is reached in 12 hrs. The oil in 50 c.c. of the ethereal solution is determined by evaporation.

CHEMICAL ABSTRACTS.

Determination of the oil content of seeds etc. D. A. COLEMAN and H. C. FELLOWS (U.S. Dep. Agric., Tech. Bull., 1928, No. 71, 1–13).—A study of Wesson's refractometric method. The method is suitable when more than 8% of oil is present. CHEMICAL ABSTRACTS.

Emulsification of sulphonated oils. D. WOODROFFE and F. N. CRANE (J. Soc. Leather Trades' Chem., 1928, 12, 419–423).—The *p*_H value of commercial products varied from 1 to 9. Some commercial products emulsified in buffer solutions over a wide range of *p*_H values (2.2–10.5), whilst others had a very limited range (8.6–10.0 and 7.8–12.0). Some, again, emulsified better in large volumes of water than in small, whereas with others the opposite was the case.

Most sulphonated oils will not emulsify in salt solutions stronger than 3%. D. WOODROFFE.

Determination of "total fatty matter" in sulphonated oils. K. REITZ (Chem. Umschau, 1928, 35, 270–274).—The available methods for the determination of "total fatty matter" of sulphonated oils are discussed, and a scheme for the collection of further data from manufacturers, analysts, etc. is suggested.

E. LEWKOWITSCH.

See also A., Nov., 1172, **Cholesterol and its effect on fatty acid films** (ADAM and JESSOP). 1176, **X-Ray investigation of long-chain compounds** (MÜLLER). 1196, **Nickel catalyst for continuous hydrogenation** (SVIZUIN). 1270, **Micro-determination of fats in blood** (MILROY).

Washing nickel catalyst. SOSENSKI.—See I. **Oil varnishes.** KRAUZ and HRACH.—See XIII. **Extracts containing fat.** HÜBSCHER. **Mercury ointment.** DIETZEL and SEDLMAYER.—See XX.

PATENTS.

Production of sulphonated oils and fats with a high content of organically combined sulphuric acid. H. FLESCH (B.P. 284,206, 4.8.27. Ger., 24.1.27. Addn. to B.P. 282,626; B., 1928, 456).—In the process of the prior patent the glacial acetic acid is replaced by other low aliphatic acids in an anhydrous condition, e.g., formic, propionic, butyric, and lactic acids, or by their acyl chlorides.

H. ROYAL-DAWSON.

Manufacture of esters of fatty acids and of mixtures containing fatty acids. E. WECKER (B.P. 273,276, 13.6.27. Ger., 23.6.26).—The removal of alcohols from difficultly volatilisable esters, after processes of esterification involving the use of excess of alcohol (e.g., glycerin), is effected by using approx. 90% of the theoretical amount of glycerin necessary for complete esterification and then removing the excess of acid by the process of B.P. 213,267 (B., 1925, 619).

E. HOLMES.

Treatment of cashew nut-shell oil and products obtained thereby. HARVEL CORP., Assees. of M. T. HARVEY (B.P. 272,510, 7.6.27. U.S., 8.6.26).—The oil is heated at about 140° with a metal, e.g., copper, aluminium, or lead, or with a metal oxide, hydroxide, or carbonate, and any residue is subsequently removed from the oil.

L. A. COLES.

Products from cashew nut-shell oil (B.P. 272,509).—See IV.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Drying of dammar oil varnishes. C. K. KRAUZ and V. F. HRACH (Chem. News, 1928, 137, 257–260).—Mixtures of equal parts of dammar resin and various oils (linseed, hempseed, locustseed, soya bean, poppyseed, and walnut) with varying amounts of driers were heated at definite temperatures for varying lengths of time, and the varnishes so obtained were examined for drying properties. The varnishes were the thinner the higher was the temperature at which they were prepared, whilst at the same temperature cobaltic oxide produced the thinnest and cobaltous oxide the thickest products. The rate of drying increased with the temperature of

heating up to a definite optimum, and again decreased as the temperature was raised further. The optimum temperatures were situated between 175° and 275°, and the same drier usually required the same optimum temperature whatever oil was used. The iodine value of the original oil is only an approximate index of the rate of drying of the varnishes, locustseed oil varnishes drying more quickly than linseed oil varnishes. The acid and saponification values decreased with rise of temperature, duration of heating, and amount of drier added. The optimum duration of heating was usually the shorter the higher the temperature used. The drier had a profound influence on the rate of drying; cobaltous oxide was most efficient for oils of high iodine value, followed by manganous oxide, lead peroxide, red lead, cobaltic oxide, and lead monoxide, but with oils of low iodine value lead monoxide was best and cobaltous oxide worst, whilst manganous oxide was the best drier for poppyseed oil varnishes and one of the worst for walnut oil. The optimum amount of drier varied considerably, being usually the greater the higher was the optimum temperature. The scraping test was applied to all varnish films, those prepared at lower temperatures being brittle whilst those prepared at the optimum temperatures were smooth and resistant.

S. COFFEY.

Oil-soluble phthalic- and phenolic-type synthetic resins. H. A. GARDNER (Amer. Paint & Varnish Manufs.' Assoc. Circ. No. 339, Oct., 1928, 673—709).—A *résumé* of patent literature on the production of oil-soluble phenol-formaldehyde and "glyptal" resins. The types are discussed generally and the results of accelerated roof tests of oil varnishes containing them are tabulated and illustrated by photomicrographs.

S. S. WOOLF.

Cellulose esters and ethers. GARDNER and KNAUSS.—See V.

PATENTS.

Paint or waterproofing material and its production. F. W. McRAE, Assr. to McRAE PAINT PRODUCTS, INC. (U.S.P. 1,684,593, 18.9.28. Appl., 13.3.28).—The light oils are evaporated from a Trinidad asphalt and a cold petroleum distillate is added, whereby the inorganic constituents are permanently suspended and the other constituents dissolved and rapidly cooled. A pigment is finally added.

F. G. CLARKE.

[Production of bituminous] paints. S. FOWLER and E. EDSEER (B.P. 298,256, 8.6.27).—The paint comprises a solution in a volatile solvent of bitumen and neutral wool fat free from soap, prepared, *e.g.*, as described in B.P. 253,995 (B., 1926, 759), with or without the addition of pigments, *e.g.*, red iron oxide.

L. A. COLES.

Production of blanc fixe. P. KIRCHEISEN (G.P. 450,180, 13.3.26).—Barium sulphate is precipitated by the action of sodium sulphate on barium sulphide solution under pressure, or is heated under pressure with sodium sulphide solution or with water after precipitation, so that it is obtained in a granular form which filters readily.

L. A. COLES.

Manufacture of white lead by electrolysis. R. S. CARRERAS (B.P. 298,520, 7.7.27. Addn. to B.P. 277,723;

B., 1927, 917).—A single tank is used in the prior process, the electrolyte comprising a dilute solution of, *e.g.*, sodium chlorate as catalyst charged with carbon dioxide. The exhausted electrolyte can be withdrawn in a small stream and replaced by fresh electrolyte at the same rate without removal of the lead carbonate. The electrodes are spaced far enough apart to allow the lead carbonate to settle as a fine sludge.

L. A. COLES.

Products from cashew nut-shell oil (B.P. 272,509).—See IV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Effect of flour in sulphur-vulcanised rubber mixtures. R. DITMAR (Gummi-Ztg., 1928, 43, 191).—Flour of various sorts aids the plastication of rubber and, although not suitable for incorporation in material to be vulcanised with sulphur chloride, can be used satisfactorily in rubber to be vulcanised with sulphur at fairly low temperatures, *e.g.*, at 100°, with the aid of an ultra-accelerator. Relative to common mineral ingredients, the tensile properties of the product are affected somewhat adversely by flour.

D. F. TWISS.

Absorption of vapours by rubber. S. REINER (Kautschuk, 1928, 4, 210—215).—Apparatus is described suitable for the measurement of the rate of absorption of vapour, *e.g.*, of petrol or benzene, by rubber. With benzene vapour at 25° progressive absorption was observed for 700 hrs. without any sign of equilibrium.

D. F. TWISS.

Evaluation factors for rubber vulcanisates. W. ESCH (Kautschuk, 1928, 4, 215—222).—A discussion of the influence of the composition of vulcanised rubber mixtures and the nature of the components on the desirable mechanical characteristics of the product.

D. F. TWISS.

Determination of inorganic matter in soft rubber goods. S. MINATOYA, H. OKUHARA, and S. OHKI (Res. Electrotech. Lab., Japan, 1928, No. 234, 42 pp.).—The finely-divided rubber (1 g.) is heated with molten paraffin wax (10 g.) at 180° until the dispersed mineral matter begins to coagulate and the upper layer of liquid clears. While warm, 70 c.c. of light petroleum are added and the mixture is centrifuged for a few minutes at 2500 r.p.m. After decanting, the residue is washed with light petroleum and again decanted. Then a mixture of equal weights of acetone and chloroform is added, and the mixture after boiling is again centrifuged. Finally the liquid is decanted, washed three times more, dried, and weighed.

D. F. TWISS.

See also A., Nov., 1186, **Micellar structure of caoutchouc** (MARK and VON SUSICH). 1252, **Caoutchouc** (MEYER and MARK).

PATENTS.

Direct production of rubber articles from aqueous dispersions of rubber and the like vegetable resins or compounds thereof. P. KLEIN, A. SZEGVARI, R. F. MCKAY, C. HAYES, and G. W. TROBRIDGE (B.P. 297,780 and 298,117, 26.3.27).—(A) The nature of electrophoretic deposits from aqueous dispersion of rubber or similar substances on a metal such as zinc is favourably influenced by the introduction of hydroxyl-free electrolytes

such as an alkali chloride, calcium chloride, or an alkali ferrocyanide. The proportion of hydroxyl ions in the dispersion may even exceed the limit of alkalinity at which, before the introduction of the said electrolyte, anodic gassing would occur. Unrequired electrolytes in the dispersion may be removed by dialysis. (B) In the production of articles from aqueous dispersions whether natural or artificial, *e.g.*, of rubber (compounded or otherwise), by a process in which the thickness of the article formed on a deposition foundation increases with the time of the operation, the surface of the article not in contact with the deposition foundation is subjected to a coagulating agent, such as moist air, steam, hot water, or a coagulant liquid or vapour before the final drying operation. This treatment improves the uniformity of the deposit and prevents fissuring during the subsequent drying. A deposit after such treatment may be coated with a fresh deposit, the procedure being repeated. D. F. TWISS.

Direct production of rubber goods. ANODE RUBBER CO., LTD. From W. C. GEER, B. DALES, and B. F. GOODRICH CO. (B.P. 297,850, 28.3.27).—In the production of articles from aqueous dispersions of rubber and/or of rubber-like substances and vulcanising agents by direct deposition on moulds or formers, ultra-accelerators, *e.g.*, diethylamine diethyldithiocarbamate, mercaptobenzthiazole, or a dithiofuroate, are included in the dispersions. Complete vulcanisation can then be effected while the articles are still on their formers, *e.g.*, in hot air or hot water, during their drying or by the time they are dried. D. F. TWISS.

Manufacture of articles from aqueous dispersions containing rubber, gutta-percha, balata, and similar resins. DUNLOP RUBBER CO., and D. F. TWISS (B.P. 297,911, 25.7.27).—Non-porous moulds or formers having on their surfaces films of coagulant liquids such as alum or formic acid are dipped in concentrated aqueous dispersions of rubber or the like, *e.g.*, into concentrated latex, containing also rubber-compounding ingredients. D. F. TWISS.

Manufacture of rubber compositions. GOODYEAR TIRE & RUBBER CO., Assees. of L. B. SEBRELL (B.P. 287,445, 11.1.28. U.S., 19.3.27).—The condensation product of crotonaldehyde and an amine, *e.g.*, aniline, formed particularly in an acidic medium, is applied as an anti-oxidant. D. F. TWISS.

Manufacture of rubber filaments and the like. M. DRAEMANN (B.P. 297,912, 25.7.27).—Rubber solution, prepared without destroying the nerve of the rubber, is extruded from nozzles, *e.g.*, by means of a plunger acting in a cylinder, the dimensions of the filaments being adjustable by the tension as they fall from the nozzles or while they are being conveyed through the successive stages. The issuing filaments are dusted with talcum powder and, after drying, are vulcanised, *e.g.*, by passage through a bath of molten sulphur. Uniform tension on all the filaments may be ensured by causing these to adhere together, *e.g.*, by means of liquid sulphur, casein, or glue applied before or after the vulcanisation stage. D. F. TWISS.

Production of a hydrogenated derivative of rubber. SOC. CHEM. IND. IN BASLE (Swiss P. 121,111,

22.3.26).—A product suitable for use in the manufacture of impregnating agents, lacquers, adhesives, etc. is prepared by masticating rubber in the absence of air or in the presence of hydrogen, and subsequently hydrogenating it in the presence of a catalyst. L. A. COLES.

Colouring of rubber. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 298,364, 6.10.27).—Calcium benzeneazo-2-hydroxynaphthalene-6-sulphonate withstands cold and hot vulcanisation and satisfactorily replaces antimony sulphide as an orange-red colour for rubber. D. F. TWISS.

Jelutong product and its manufacture. S. S. YATES, ASSR. to CHICLE DEVELOPMENT CO. (U.S.P. 1,685,797, 2.10.28. Appl., 24.9.27).—Jelutong latex is coagulated, melted, heated until the water content is below 5%, and cast into moulds. F. G. CLARKE.

Treatment of latex and products obtained therefrom. U.S. RUBBER PLANTATIONS, INC., Assees. of W. A. GIBBONS and J. MCGAVACK (B.P. 290,242, 19.3.28. U.S., 11.5.27).—See U.S.P. 1,673,672; B., 1928, 616.

Insulating materials (B.P. 298,694).—See XI.

XV.—LEATHER; GLUE.

Osage-orange and fustic extracts. G. DESMURS (J. Soc. Leather Trades' Chem., 1928, 12, 424—429).—Extracts of liquid Cuba fustic and of liquid and dry osage-orange, respectively, had *d* 1.280, 1.255, 4.9; solid matter 55.7, 56.9, 95.1; soluble matter 54.3, 50.8, 85.2; non-tans 16.3, 10.7, 19.0; tans 38.0, 40.1, 66.2; insoluble matter 1.4, 6.1, 9.9%; colour of 0.5% solution, red 3.2, 4.8, 6.2; yellow 24.8, 26.8, 26.0. The extracts can be differentiated by the orange-coloured filtrate of fustic in the formaldehyde-hydrochloric acid reaction (*cf.* "Leather Chemists' Pocket Book," 1919, 51) and the yellow filtrate with osage-orange. A blue or violet coloration indicates the presence of quercitrin, chestnut, myrobalans, or other pyrogallol tannin. The precipitates obtained with lead acetate-acetic acid are: osage-orange (yellow), fustic (orange); and with sodium hydroxide-lead acetate are: osage-orange (orange), fustic (yellow). On Mulhouse strips, fustic and osage-orange give the following colours, respectively, on wool, reddish-beige, dull yellow; wool mordanted with stannous chloride, dull golden-yellow, bright golden-yellow; mordanted with alum, brownish-yellow, dull yellow; mordanted with alum and stannous chloride, orange-yellow, golden-yellow. Cotton dyed with osage-orange shows a deep yellow colour by a Wood light and fustic a slightly mauve-tinted yellow. 3% of fustic extract and 0.1—0.15% of potassium titanium oxalate is recommended for dyeing tan or yellow shades. It can be used for bottoming chocolate, champagne, blood-red, or dark olive-green shades, and various shades of brown and maroon on shoe leathers, gloving, or chamois leathers. In textile dyeing it is useful in correcting the bluish tone of logwood-blacks, in yellowish-beige shades, for bottoming brownish shades, greens, olives, and for mordanting cotton.

D. WOODROFFE.

Tannin of *Geranium maculatum*. J. C. and B. L. DE G. PEACOCK (Amer. J. Pharm., 1928, 100, 548—557;

cf. B., 1927, 790).—The powdered drug, examined by methods previously described, behaved very similarly to *Heuchera Americana*. The tannin of *G. maculatum* is not like gallotannic acid, and does not give either gallic acid or dextrose on decomposition by mineral acids. Gallic acid is not present in the drug and is not formed on drying the rhizome, but the latter contains a crystalline acid substance which gives a green colour with ferric chloride and which decomposes the soluble tannin with formation of phlobaphen. A white, water-soluble tannin is the primary source of the various forms of phlobaphen found in the commercial drug and its preparations.

E. H. SHARPLES.

Tintometer [for measuring the colour of tan liquors]. J. G. PARKER (J. Soc. Leather Trades' Chem., 1928, 12, 417—418).—A modified Lovibond tintometer is described in which there is only one eyepiece. The glasses are fixed on movable, horizontal carriers which are controlled by sliding knobs on the front of the apparatus. The units and decimals are in separate rows. By sliding the glasses backwards and forwards, a perfect colour match is obtained more speedily than with the old tintometer.

D. WOODROFFE.

Commercial gelatin and glue. M. RÜDIGER and E. MAYR (Kolloid-Z., 1928, 46, 81—89).—The chemical compositions and physical properties of 7 kinds of commercial gelatin have been determined, and the results indicate that chemical analysis furnishes little or no evidence as to the properties of a sample of gelatin, the important factor being the content of glutin. Herold's method of determining glutin is applicable to leaf gelatin, but gives inconsistent results with gelatin containing a high percentage of degradation products. Titration of gelatin solutions with formaldehyde solutions does not give quantitative results as to the amount of decomposition products present, but serves as a rough indication. Determination of glutin gravimetrically in the residue after washing out the degradation products gives consistent results and serves as a means of evaluating a sample of gelatin. Trunkel's method of determining gelatin by addition of tannin depends on the glutin content. The initial viscosity and the change of viscosity with time are dependent on the glutin content and afford a qualitative means of characterisation. The surface tension of a gelatin solution is independent of the amount of gelatin, and is determined mainly by the presence of impurities.

E. S. HEDGES.

Pigments of cacao beans. FINCKE.—See XIX.

PATENTS.

Unhairing and/or softening animal skins. SOC. PROGIL (B.P. 284,719, 3.2.28. Fr., 4.2.27).—Skins are treated with water containing the residuary liquors from cultures of various bacteria, e.g., *B. subtilis*, *B. mesentericus*, *B. liquefaciens*. The liquor must be at 37°, and additions of 0.002% of ammonium chloride may be made for softening, or of sodium bicarbonate for unhairing and softening. The wool or hair is loose in a few hours. [Stat. ref.]

D. WOODROFFE.

Preparation of protein colloids [adhesives, size, etc.]. C. ARNOLD. From I. F. LAUCKS, INC. (B.P. 298,511, 28.3.27).—Vegetable protein, e.g., ground

soya-bean flour, is treated with a compound containing sulphur linked to carbon, but not to oxygen, e.g., aliphatic or aromatic sulphides or disulphides, or alkali or alkaline-earth thiocarbonates, with or without the addition of alkali or alkaline-earth compounds, e.g., sodium or calcium hydroxide or sodium fluoride, and agents to retard the setting time. [Stat. ref.]

L. A. COLES.

Composition for preserving and curing hides and skins. H. DODGE (B.P. 298,435, 20.2.28).—See U.S.P. 1,680,136; B., 1928, 763.

XVI.—AGRICULTURE.

Report of the Agricultural Research Station at Halle. F. MÜNTER (Landw. Jahrb., 1928, 67, 1—102; Bied. Zentr., 1928, 57, 403—409).—Field trials with rye and potatoes on light sandy soils showed the value of farmyard manure used in conjunction with artificial fertilisers, its value being greater as an ameliorative factor than as a nutrient. Crop increases following the joint use of yard manure and artificials were greater than the sum of the increases resulting from their separate use. The increased assimilation of mineral fertilisers resulting from the simultaneous use of farmyard manure is most marked in the case of potassium. In green-manuring trials yellow lupins produced the largest increase in soil organic matter, and the greatest nitrogen accumulation. The efficiency of green manures depended more on favourable climatic conditions than on their organic and nitrogen contents. Occasional use of yard manure increased the efficiency of green manures. Physiologically acid fertilisers increased the assimilation of phosphates by crops, and subsequent liming retarded or eliminated this effect. Light top-dressings of nitrogenous fertiliser on legumes produced greater initial growth and increased resistance to bad climatic conditions and fungoid diseases. No stimulation of growth of sugar beet by iodine was observed.

A. G. POLLARD.

"Single-value" soil properties: significance of certain soil constants. B. A. KEEN and J. R. H. COURTS (J. Agric. Sci., 1928, 18, 740—765).—A number of single-value soil determinations are discussed, and comparison is made of values obtained with natural soil samples and the same after treatment with hydrogen peroxide. The pore space of oven-dried, kneaded blocks of soil approximated to a mean value of 26% in all cases examined. This figure was reduced by 4—5% by treating the soil with hydrogen peroxide, which removes 75% of the soil organic matter. Correlation of various pairs of values obtained showed heavy clay soils to be characterised by high ignition losses, moisture contents, and "sticky points." Indications were obtained that the "sticky-point value" is controlled by the content of colloidal organic matter and clay. The moisture content of a soil at 50% R.H. was largely controlled by the actual clay content. The "sticky-point" value approaches a lower limit of 16% in sandy soils containing little organic matter. The approximate distribution of soil moisture as between organic and inorganic colloids is discussed.

A. G. POLLARD.

Proteins of different types of peat soils. W. L. DAVIES (J. Agric. Sci., 1928, 18, 682—690).—Wet peat

deposits showed a greater degree of humification than dry deposits. Of the total nitrogen in peats, boiling hydrogen peroxide extracted 70–80%, and of this 60–70% was obtained as ammonia. Hydrochloric acid (constant-boiling mixture) extracted a similar amount of nitrogen consisting of ammonia, humin, mono- and di-amino-acids. Peat proteins yielded 3–5 times as much amide nitrogen as do pure animal or vegetable proteins. Protein decomposition in soil is reflected in the ratio of mono- to di-amino-acid nitrogen, which increases with protein degradation. Dry peats showed a narrower ratio. The presence of calcium bicarbonate and of moving water aids the decomposition of peat protein by removal of the products. Extracts of peat with 2.5% potassium hydroxide solution contain less nitrogen than the acid extracts, but the proportion of mono- to di-amino-acid nitrogen is similar. Soil conditions favouring humification also favour protein decomposition.

A. G. POLLARD.

Integral dissociation of silicates by carbonic and humic acids [in soils], and succeeding reactions. R. GUILLIN (Compt. rend., 1928, 187, 673–675).—Carbonic acid acts on complex silicates to eliminate the alkalis with the formation of argillaceous silicates, and the subsequent breakdown of these has been investigated by a study of the compositions of different humiferous earths. These may be divided into two classes—non-calcareous and calcareous. The non-calcareous earths contain a high proportion of lime and iron oxide, but these are not combined with humic acids, which are united only to the alumina. The iron on liberation from the silicate is reduced to the ferrous state, but the alumina can be liberated only by the decomposition and combustion of the humates. In the calcareous humiferous earths more lime and less alumina are united with the humic acids as the earth becomes richer in limestone and poorer in silicates. It is concluded that whether the resulting earths are calcareous or not, carbonic and humic acids can cause the integral dissociation of rocks, with the elimination successively of potash, soda, lime, and magnesia, and finally of alumina which remains united with the humic acid, the iron being removed by reduction.

W. HUME-ROTHERY.

Effect of colloidal silica on the [fertilising] efficiency of phosphates. S. SEKI (Bul. Sci. Fak. Terkult. Kjusu, 1928, 2, 253–261).—In sand-culture experiments with rice, the addition of colloidal or precipitated silica markedly increased the efficiency of tricalcium phosphate, and slightly increased that of acid phosphate. The amount of ash and silica in the straw and in mixtures of husk and chaff increases with the supply of silica, as do also the amount of phosphate, and its percentage of total plant-phosphate, in the unhulled rice.

CHEMICAL ABSTRACTS.

Oxidation of sulphur in limed and unlimed soils. O. M. SHEDD (Soil Sci., 1928, 26, 93–105).—Sulphur oxidation was little influenced by liming as adjudged by experiments on moist bottled soils. Very little consistent relation was found between the hydrogen-ion concentrations of the treated soils, either in their initial or final p_H values and their acidity or alkalinity

by titration or in the amounts of added sulphur oxidised by them. Increased aeration resulted in increased oxidation.

H. J. G. HINES.

Application of the antimony electrode to the determination of p_H values of soils. E. F. SNYDER (Soil Sci., 1928, 26, 107–111).—From a comparison between the values obtained on eight soils it is concluded that substantially the same results are obtained with the antimony as with the hydrogen electrode.

H. J. G. HINES.

Revised official British method for mechanical analysis [of soils]. SUB-COMMITTEE OF THE AGRIC. EDUCATION ASSOC. (J. Agric. Sci., 1928, 18, 734–739).—The standard temperature for sedimentation experiments adopted is 20°. Fractions of soil are weighed in the oven-dried (100–105°) condition and not ignited as heretofore. The number of fractions is reduced to four, and the sampling depths and times are modified. The new scale includes (1) clay, settling 10 cm. in 18 hrs.; (2) silt, 10 cm. in 4 min. 48 sec.; (3) fine sand with maximum diam. 0.2 mm.; and (4) coarse sand with maximum diam. 2.0 mm. Fractions (3) and (4) are separated with sieves. The sieve used for the fine sand fraction is the standard No. 70 I.M.M. sieve.

A. G. POLLARD.

Mechanical analysis of soils. M. KÖHN (Landw. Jahrb., 1928, 67, 485–546; Bied. Zentr., 1928, 57, 385–386).—Standard methods for the mechanical analysis of soils are discussed from the point of view of their limitations and sources of error. Soil particles of diameter greater than 0.02 mm. can be satisfactorily fractionated by means of sieves; sedimentation methods are applicable only to particles of less diameter than 0.05 mm. Apparatus is described for the pipette method, in which the 10-c.c. pipette used for withdrawing the sample is fitted with a three-way stopcock and supported on a frame which can be raised or lowered on a ratchet attachment without undue vibration. The pipette is filled to above the mark and the excess of suspension is rinsed out through a side tube from the stopcock. The sample and rinse water is delivered through the third side tube directly to the vessel in which it can be evaporated.

A. G. POLLARD.

Mitscherlich's law of crop growth. A. RIPPEN [with B. LEHMANN and A. STORCK] (Z. Pflanz. Düng., 1928, A12, 38–55).—Pot experiments with oats, mustard, and sunflowers, using on the same soil a rising series of (a) nitrogen dressings, (b) potash dressings, led to the following conclusions: The final yield varies according to the plant and is not a constant for a given nutrient, as Mitscherlich supposes. The Mitscherlich law could not be applied to any physiological characteristics such as time of flowering or time of ripening. It is concluded that a simple equation of the Mitscherlich type cannot account for the observed facts of growth.

H. J. G. HINES.

Crop variation. V. Relation between yield and soil nutrients. B. BALMUKAND (J. Agric. Sci., 1928, 18, 602–627).—Mathematical relationships between the growth of plants and the amount of nutrient material supplied are discussed. Formulae analogous to those expressing the relationships of electrical resist-

ance show close correlation with experimental data and are of practical value in that they express the results of variations of two independent factors simultaneously and yet contain constants largely independent of environmental conditions. Such mathematical representations are much more closely in agreement with experimental records than those put forward by Mitscherlich.

A. G. POLLARD.

Growth of cotton in various nutrient solutions.

H. J. HARPER and H. F. MURPHY (Soil Sci., 1928, 26, 139—145).—Several well-known nutrient solutions were tested in an endeavour to ascertain the best media for the growth of cotton in sand cultures. Of those tried, Tottingham's solution was the most satisfactory.

H. J. G. HINES.

Nitrification and its relation to crop production on Carrington loam under different treatments.

L. W. ERDMAN and H. HUMFELD (Iowa Agric. Exp. Sta. Res. Bull., 1928, No. 110, 262—291).—Crop rotation did not appreciably affect the nitrifying power of untreated soils or those treated with ammonium sulphate, ammonium sulphate and calcium carbonate, or crop residues and lime. The nitrifying power as measured by ammonium sulphate, the reaction after thirty days' incubation, and the crop yield are related, but not perfectly when calcium carbonate is present. The presence of lime increased the nitrifying power of soils. Soil reaction and soil nitrifying power are related. Soil moisture was not affected by crop rotation or soil treatment.

CHEMICAL ABSTRACTS.

Action of lime in the rendering available of soil nutrients. J. SOUČEK (Z. Zuckerind. Czechoslov., 1928, 53, 101—104).—In pot experiments with oats a dressing of lime applied 3 weeks before sowing produced a considerable increase in the assimilable nitrogen of soils, but had no notable effect on the availability of potassium and phosphorus.

A. G. POLLARD.

Selective absorption of inorganic elements by various crop plants. J. D. NEWTON (Soil Sci., 1928, 26, 85—91).—Sunflowers, beans, peas, barley, wheat, and maize were grown together in carefully controlled water and soil cultures and showed differences and similarities characteristic of the species studied.

H. J. G. HINES.

Neubauer seedling method [for determining plant nutrients in soil]. W. KROSS (Landw. Jahrb., 1928, 67, 629—662; Bied. Zentr., 1928, 57, 409—411).—To prevent the caking of surface soil in vegetation experiments, broken porcelain (2—4 mm.) was added. By increasing the proportion of soil or sand, it was found that the intake of phosphorus by plants was not proportional to the amount of soil phosphate used. Comparison of the assimilation of nutrients in fertilised soils showed the influence of existing soil potash and phosphate to be very small. There were considerable differences in the intake of easily soluble phosphates. The effect of nitrogenous fertilisers on the assimilation of tertiary calcium phosphate was very marked and was ascribed to chemical changes induced in the nutrient medium. The intake of phosphorus by plants supplied with calcium phosphate was not proportional to the amounts of phosphate added. Increasing quantities of

easily soluble phosphates used in conjunction with a basal fertiliser were never completely utilised by plants. Control experiments with sand and mineral salts are recommended. The "assimilation values" in seedling experiments are influenced by the excess of nutrients present. Experimental values obtained by the Neubauer method are not directly applicable to field work, and there is insufficient information as to variations in nutrient requirement and assimilative capacity of the roots among different crops. The Mitscherlich method for determining soil nutrient values is preferred.

A. G. POLLARD.

Certain acid soils and growth of sugar beet.

G. NEWLANDS (J. Agric. Sci., 1928, 18, 704—712).—Variations in the yield of sugar beet were compared with differences in mechanical analyses, p_H values, lime requirements, easily extractable calcium (0.05N-hydrochloric acid), and humified matter of a number of soils. No correlation was observed between crop yield and mechanical analysis. In soils with p_H less than 5.3 and easily extractable calcium less than 0.12%, growth of beet was poor or failed. In the range p_H 5.3—6.2 growth was relatively good. In soils of different type there was no relationship between the lime requirement and other values examined, but in any one soil type changes in p_H value, lime requirement, and extractable calcium were comparable and in accord with crop yields. In soils with similar contents of easily extractable calcium those of higher humus content had the higher lime requirements. In a more detailed examination of different areas of one soil, the values for p_H , lime requirement, exchangeable calcium, titratable acidity, and degree of saturation correlated well with each other and with the crop yields of beet.

A. G. POLLARD.

See also A., Nov., 1205, **Calcimeter for gasometric determination of calcium carbonate** (HOCK). 1289, **Prolonging the germinative faculty of seeds** (GUILLAUMIN). **Angiosperm seeds and germination** (NIETHAMMER). **Nutrition of plants with aldehydes** (SABALITSCHKA). 1290, **Relation of boron to growth of tomato plants** (DORE). **Influence of sulphur on nitrogen and phosphoric acid in plants** (KALUSHSKI). **Nitrate reduction by plant roots** (SCHMUCK). **Effect of light on nitrate assimilation in wheat** (TOTTINGHAM and LOWSMA). 1291, **Influence of temperature on growth of fungi** (MEYER). **"Lead disease" of plants** (ALBIZZATI). **Spike disease of sandal** (IYENGAR).

Ionic exchange in zeolitic silicates. KAPPEN and FISCHER.—See VII. **Manganese content of vegetables.** PETERSON and LINDOW.—See XIX.

PATENTS.

Production of fertilisers. M. P. NITSCHKE (U.S.P. 1,684,712, 18.9.28. Appl., 25.9.26. Ger., 8.10.25).—An acid sulphate is added to waste sulphite-cellulose lye, and the mixture evaporated to remove sulphur dioxide.

F. G. CLARKE.

Production of mixed manures. RHEINANIA-KUNHEIM VER. CHEM. FABR. A.-G. (B.P. 283,194, 15.11.27. Ger., 8.1.27).—Ammonium salts are mixed, in the dry condition, with calcined phosphates, the alkalinity of

which is subsequently neutralised by the addition of acids or acid salts (e.g., triammonium monohydrogen disulphate, sulphuric acid, etc.). Other fertiliser salts may also be added.

A. G. POLLARD.

XVII.—SUGARS; STARCHES; GUMS.

See A., Nov., 1186, Micellar state of starch (MALFITANO and CATOIRE). 1189, Transition between glassy and liquid states of dextrose (PARKS, HUFFMAN, and CATOIR). 1191, Rôle of phosphates in oxidation of dextrose (KAPPANNA). 1213, Reactions relating to carbohydrates and polysaccharides (HILL, WHELEN, and HIBBERT). Mechanism of the acetal reaction (HILL and PIDGEON). 1219, Colour tests for sugars (WONG). 1220, Colour reactions of carbohydrates (EKKERT). Rotatory dispersion of sugars (WAGNER-JAUREGG). Degradation of dextrose by oxidation (BLEYER and BRAUN). 1222, Mechanism of carbohydrate oxidation (EVANS and O'DONNELL). 1223, Sucrose *B* (PICTET and VOGEL). Sucroses *C* and *D* (PICTET and VOGEL). 1224, New sugars of trehalose type (VOGEL and DEBOWSKA-KURNICKA). Sugar syntheses (HELPERICH and BREDERECK). Synthesis of raffinose (VOGEL and PICTET). Röntgen diagram of starch (VON NÁRAY-SZABÓ). 1225, Potato starch (HESS, FRIESE, and SMITH). Inulin (PRINGSHEIM and REILLY). 1281, Hydrolysis of sucrose by enzymes (WEIDENHAGEN). Enzymic decomposition of dextrose to lactic acid (BRUNUS and PROFFE). 1284, Fermentation of sugar (KOBEL and TYCHOWSKI). 1285, Trehalosemonophosphoric ester from fermented sugars (ROBISON and MORGAN). Alcoholic fermentation of sugars (NEUBERG and SIMON).

Starch content of potatoes. PAROW; also von SCHEELE and SVENSSON. Dextrins from honey and artificial honey. FIEHE and KORDATZKI.—See XIX.

PATENTS.

Recovery of sucrose from mixtures containing reducing sugars. H. W. DAHLBERG (U.S.P. 1,686,440, 2.10.28. Appl., 23.9.26).—Mixtures containing at least 2.8 pts. of sucrose per 1 pt. of reducing sugar are treated with an alkaline-earth metal to precipitate the sucrose as its saccharate, and this is removed and decomposed into an alkaline-earth compound and sugar syrup.

L. A. COLES.

Glueing wood with starch or starch-containing substances. J. FLASCHNER (B.P. 279,087, 12.10.27. Ger., 14.10.26).—Starch etc., alone or mixed with other substances, is applied to the wood surface at the ordinary temperature, and then heated at 90° under pressure.

L. A. COLES.

XVIII.—FERMENTATION INDUSTRIES.

Substitution of ammonium salts for malt combs in the preparation of yeast. H. CLAASSEN (Z. angew. Chem., 1928, 41, 1161—1163).—The work of Wohl and Scherdel (B., 1921, 191 A) is unsatisfactory in that the calculated ratios of organic to ammoniacal nitrogen are based on the assumption that all the nitrogen of malt extracts is assimilable by yeast. Actually only 65% of

this nitrogen has any nutrient value. With the optimum concentration of assimilable nitrogen the replacement of organic nitrogen by ammonium salts up to 40% does not markedly affect the yield or nitrogen content of the yeast. When more than 40% of ammoniacal nitrogen is used the yield decreases and the nitrogen content of the yeast increases. With three times the optimum amount of nitrogen in the nutrient medium, the substitution of ammonium salts results, in all cases, in a decreased yield and higher nitrogen content of yeast, although the changes are irregular. Baking qualities of the yeast decline as a result of the use of excessive amounts of ammonium salts.

A. G. POLLARD.

Method of brewing with flaked potatoes. E. LÜHDER (Z. Spiritusind., 1928, 51, 289—290).—According to the method of heating, from 140 to 170 litres of water per cwt. of flaked potatoes are heated at 52—55° in the pre-mash vessel with the necessary amount of green malt for liquefaction and 2—4% of the flaked potatoes. The alkalinity of the mash may be corrected by the addition of sulphuric acid. After the gradual addition of the remainder of the flakes, the temperature is raised to and retained at 77—79° for 15 min. with constant stirring to facilitate liquefaction. When the mash is cooled to 61° the gradual addition is begun of the malt necessary for saccharification, the last portions being added at 56—57°, and the whole, after being energetically stirred for 15 min., allowed to remain for 45 min. The yeast is introduced at 29°, and at 22° the mash is pumped to the fermentation vessels. The longer time required for the mashing operations with flaked potatoes can be curtailed by mashing with a mixture of raw and flaked potatoes. C. RANKEN.

Determination of fusel oil in alcoholic fluids [low-grade wines]. W. KILP and B. LAMPE (Z. angew. Chem., 1928, 41, 1163—1164).—Röse's method is not applicable to large amounts of fusel oil owing to the separation of the oil on dilution with water. To eliminate this the original method is modified by using aliquot portions of the test liquor and diluting with ethyl alcohol free from amyl alcohol. The further dilution with water in the requisite proportion to give 24.68% by wt. may safely be made.

A. G. POLLARD.

Radioactivity of wines. A. NODON and G. CUVIER (Compt. rend., 1928, 187, 725—727).—Various red and white wines have been found to exhibit radioactivity of the order of 0.1—0.01 of that of uranium. The intensity of the radioactivity is approximately proportional to the amounts of dried extract and reducing sugars present, and inversely proportional to the alcohol content. Yearly variations occur, these being much greater for white wines than for red.

J. W. BAKER.

See also A., Nov., 1281, Specific nature of invertase (WEIDENHAGEN). Hydrolysis of sucrose by enzymes (WEIDENHAGEN). Enzymic decomposition of dextrose to lactic acid (BRUNUS and PROFFE). Succinoxidase (CLUTTERBUCK). Specificity of the dehydrases (BERNHEIM). 1282, Oxidising enzymes (ONSLow and ROBINSON). Tyrosinase of *Beta vulgaris* (WEIDENHAGEN and HEINRICH). Stalagmometric

determination of lipases (KRIJGSMAN). Lipase activity (JOHNSON). Metaphosphatase (KITASATO). 1283, Determination of pepsin (BEER and PECZENIK). Urease (JACOBY). 1284, Yeast amylase (GOTTSCALK). Fermentation of sugar (KOBEL and TYCHOWSKI). Co-zymase and its determination (MYRBÄCK). 1285, Trehalosemonophosphoric ester from fermented sugars (ROBISON and MORGAN). Alcoholic fermentation of sugars (NEUBERG and SIMON). Formation of catalase by *B. coli* (FERNÁNDEZ and GARMÉNDIA). Catalase content of bacteria (VIRTANEN and WINTER). Oxidations by *B. xylinum* (BERNHAEUER and SCHÖN). Pyocyanine in *B. pyocyaneus* (WREDE and STRACK). 1289, Enzyme content of germinating wheat (PROKOPENKO). 1291, New enzyme, glucose-oxidase (MÜLLER). Production of invertase by *Penicillium glaucum* (KERTÉSZ).

PATENTS.

Preparation of a mild, aromatic, Yoghurt-curdled milk or sweet yoghurt junket. A. ZIERER (B.P. 272,468, 19.5.27. Austr., 9.6.26).—Boiled or pasteurised milk, mixed with fruit essences if necessary, is fermented at 28–32° with a mixture of yoghurt bacteria (*Bacterium bulgaricum*), cocci which produce rennin and acid (*Micrococcus lactis acidii*, Löhnis), and certain *Torulae* and *Mycodermae*, which produce neither alcohol nor carbon dioxide. C. RANKEN.

Treatment of animal skins (B.P. 284,719).—See XV. Fodder and food (U.S.P. 1,685,004).—See XIX.

XIX.—FOODS.

Some oxidising effects of flour bleaching. E. B. WORKING (Cereal Chem., 1928, 5, 431–435).—The bleaching of flour has a similar effect on the development of the dough to that of oxidising agents (cf. B., 1928, 621). The results after 3 weeks were found to be better than those immediately after treatment. The quality of normal flour improves for some time after milling, and then begins to deteriorate. This deterioration begins sooner in bleached than in unbleached flour. It is suggested that large bakeries should prolong the period of maximum quality by buying unbleached flour and bleaching it shortly before use.

W. J. BOYD.

Effect of stage of maturity on composition and baking quality of marquis wheat. C. E. MANGELS and T. E. STOA (Cereal Chem., 1928, 5, 385–394).—Cuttings were made at the dough, hard-dough, glazed, normal ripe, and dead ripe stages. Yield and weight per bushel increased up to the glazed stage, but protein content showed no consistent variation. Ash content and diastatic activity of the patent flour decreased as the wheat matured, but sugar content remained constant. Baking tests showed slightly better quality for the mature wheat. Nitrogen soluble in 70% alcohol increased consistently with advancing maturity. Nitrogen soluble in 5% potassium sulphate solution, amino-nitrogen, and nitrogen not precipitated by tungstic acid decreased in the dough and hard-dough stages. Glutenin attained a maximum in the hard-

dough stage and then decreased right up to the extralate stage. W. J. BOYD.

How the experimental baking test [for flour] has been developed. C. L. BROOKE and R. C. SHERWOOD (Cereal Chem., 1928, 5, 366–374).—A résumé of the literature regarding the evolution of the test.

W. J. BOYD.

Determination of moisture in wheat and flour. "Moisture testing" in water-ovens and electric ovens. E. A. FISHER and C. R. JONES (J. Agric. Sci., 1928, 18, 649–670).—Absolute values of the moisture content of wheat and flour are not obtainable by ordinary oven drying, but concordant results with an experimental error not exceeding 0.01% are conveniently obtained by the method following:—The sample (5 g.) is dried in an aluminium container fitted with a lid which is removed only in the oven. Electrically-heated ovens fitted with metal thermostats are recommended. Drying should occupy at 110° 3–12 hrs. for flour and 12–15 hrs. for wheat; or at 120° 1 hr. and 4 hrs., respectively; or for wheat at 140° 1½ hrs. The oven should not be opened during the drying period. Calcium carbide is as efficient a desiccating agent as sulphuric acid. A. G. POLLARD.

Criteria of the validity of analytical methods used by cereal chemists. A. E. TRELOAR and J. A. HARRIS (Cereal Chem., 1928, 5, 333–351).—Important criteria of the validity of methods of analysis of samples of unknown composition are: (1) average yields and differences of average yields of the same constituent of wheat or flour as determined by two or more methods, (2) consistency of the results obtainable by any method applied by various workers. Formulæ bearing on these points and illustrations of their application are given. Of the three main determinations carried out on wheat and flour, viz., those of moisture, ash, and protein, the determination of protein gives the most consistent results and that of ash the least. The presence of definite personal or laboratory equations is demonstrated, proving that concordance of results is no proof of correctness. W. J. BOYD.

Critical study of some methods used in flour colorimetry. F. V. HOOFT and F. J. G. DE LEEUW (Cereal Chem., 1928, 5, 351–365).—In determining the gasoline colour value of flour the adoption of official samples of organic dyes standardised against pure carotin solution is suggested as preferable to the use of potassium chromate. If a solution of the latter is used it should be buffered (cf. Jorgensen, A., 1927, 952). Errors arise in the use of the Kent-Jones colorimeter when samples of widely different gasoline colour value are compared, probably owing to differences in the p_H value of the standard at different dilutions. There is a direct relation between the bran content (and, in general, the ash content) and the methyl alcohol value suggested by Kent-Jones and Herd (B., 1927, 762), but with the Duboseq colorimeter, which is more convenient than that of Kent-Jones, it was found more satisfactory to use a buffered solution containing 0.005% of potassium chromate as standard instead of the mixture of potassium chromate and cobalt nitrite.

The temperature of extraction has a marked effect on the result.

W. J. BOYD.

Lipoid phosphorus of wheat and its distribution.

B. SULLIVAN and C. NEAR (Cereal Chem., 1928, 5, 436; cf. B., 1928, 621).—A correction.

W. J. BOYD.

Calculation of dry weight of milk. R. SAAR (Z. Unters. Lebensm., 1928, 55, 573—577).—From Fleischmann's formula (J. Landw., 1914, 62, 167) $t = 1.2f + 2.665(100 - 100/s)$, where t is the total dry weight %, f the fat %, and s the sp. gr., is derived the formula $r = 0.25d + 0.26 + 0.2f + c$ in which r is the fat-free dry weight %, d the lacto-densimeter reading, and c a value depending on the sp. gr. of the milk. For $d = 21.7 - 23.7$, $c = -0.02\%$; for $d = 23.8 - 26.5$, $c = -0.01\%$; for $d = 26.6 - 38.4$, $c = 0.00\%$. It is thus shown that the Herz formula $r = 0.25d + 0.2f + 0.26$ gives correct results except for highly diluted milk, for which the above small correction c should be applied.

W. J. BOYD.

Potentiometric determination of chlorine in milk. C. ROHMANN (Z. Unters. Lebensm., 1928, 55, 580—590).—A reversible adsorption of chlorine ions on the colloidal protein of the milk occurs which causes the values found by potentiometric titration to be slightly low. To avoid this source of error the following procedure is adopted. The titration is carried out slowly and the potential adjusted after 10 min. by further addition of silver nitrate solution. The liquid is then boiled and cooled and the potential readjusted within 10 min. This may be repeated. At higher concentrations an irreversible adsorption of silver ions occurs even in the presence of excess of chlorine ions, which causes high values to be obtained. This source of error becomes negligible at a dilution of 1 in 9. The amount of acid added is important, but concordant results are obtained with amounts varying from 2% to 10% of 2*N*-sulphuric acid or nitric acid.

W. J. BOYD.

Determination of the f.p. of milk. P. WEINSTEIN (Z. Unters. Lebensm., 1928, 55, 590—602).—Pritzker's method (A., 1917, ii, 549) gives good results, but its usefulness depends on the choice of a suitable thermometer. It is not of great importance whether the latter has a fixed or an adjustable zero point, but the bulb should be large and not too long and narrow. In agreement with Pritzker, the air-jacket is found to be unnecessary. For mixed milk of not less than four cows the lowest value for $\Delta \times 10^2$ should be taken as 53.5. At most a 5% addition of water may thereby escape detection. The f.p. determination is equal in usefulness to the nitrate test. In cases of low refraction or fat-free dry substance it enables distinction to be made between milk which has been adjusted and milk of low value by nature. Abnormal milk from diseased udders which might appear to have received an addition of 20—30% of water has the same f.p. as normal milk.

W. J. BOYD.

Determination of moisture in dry skim milk by the Bidwell-Sterling toluene distillation method.

P. A. WRIGHT (J. Dairy Sci., 1928, 11, 240—242).—Moisture is preferably determined by distillation with

toluene (2 hrs.), which gives slightly higher results than heating in a vacuum oven. CHEMICAL ABSTRACTS.

Butter-fat tests on first and later lactations.

C. F. MONROE (Ohio Agric. Exp. Sta. Bull., 1927, No. 12, 34—38).—First-lactation milk of Holstein and Jersey cows contained, respectively, 3.4 and 5.4% of fat, later average values being within 0.1% of these; variations in individual lactations reached 0.9 and 1.1% (average difference 0.36 and 0.57%).

CHEMICAL ABSTRACTS.

Detection of thickening agents in whipped cream.

O. BAUMANN (Z. Unters. Lebensm., 1928, 55, 577—580).—If the product has been shown to be free from calcium and magnesium saccharates, it is tested for tragacanth by diluting 5 g. of sample with an equal quantity of water, adding 2 c.c. of 10% ammonia solution, and shaking in a separating funnel successively with alcohol, ether, and light petroleum, according to the method of Gottlieb and Röse. The aqueous layer is then allowed to run off and the last drops are observed. If tragacanth is present these will be ropy and viscous. For the detection of gelatin, 25 c.c. of the sample are boiled with 25 c.c. of water and 5 c.c. of lead acetate and filtered hot. If the filtrate gives a yellow precipitate with concentrated picric acid solution gelatin is present. If gelatin is absent the filtrate is tested for agar-agar by adding a 5% tannic acid solution instead of the picric acid solution. A copious precipitate indicates the presence of agar-agar.

W. J. BOYD.

Disintegration of rice straw. H. IWATA (J. Dep. Agric. Kyushu, 1926, 1, 217—240).—The dry matter of rice straw has a starch value of about 20%. The composition and nutritive value are not changed by boiling the straw in water for 3 hrs. or soaking it in 0.25% sodium hydroxide solution for 4 hrs.; soaking in 0.75 or 1.5% sodium hydroxide solution or boiling in 1% milk of lime for 3 hrs. extracts incrusting substances, increases the digestible matter, and raises the starch value of the dry matter to 53—58%.

CHEMICAL ABSTRACTS.

Evaluation of potatoes according to the starch content.

E. PAROW (Z. Spiritusind., 1928, 51, 290).—Although the yield of potato flakes increases and working costs relatively diminish when the content of starch calculated on the raw potato rises, the increase in price of the raw potato, if based on its starch content, more than counterbalances those factors. Accordingly, the manufacturers of potato flakes wish potatoes to be valued according to the starch content calculated on the dry matter in the potato, and not, as at present, according to the starch content in the raw potato.

C. RANKEN.

Determination of starch in potato pulp.

C. VON SCHEELE and G. SVENSSON (Z. Spiritusind., 1928, 51, 290—291).—If the usual methods for the determination of starch in cereals are applied to potato pulp, the results obtained are much too high owing to the presence of appreciable quantities of substances of a pectinous nature, which are soluble and give rise during the hydrolysis of the starch to optically active and reducing substances. To minimise the possible degradation of the pectins during hydrolysis, the starch should be brought to the

soluble form as quickly as possible and the pectins precipitated by lead acetate. Any excess of lead acetate is removed by sodium phosphate and the starch determined in the filtrate by the polarimeter or refractometer. Maercker's method of bringing the starch into solution by diastase, and subsequently removing the pectins by lead acetate, although rather lengthy, gives the best results. C. RANKEN.

Variations in the manganese content of certain vegetables. W. H. PETERSON and C. W. LINDOW (Soil Sci., 1928, 26, 149—153).—The manganese content of cabbages, peas, beans, and tomatoes appears to bear no relation to type of soil, variety, size or date of harvest. H. J. G. HINES.

Detection of excessive quantities of husk in cocoa and cocoa preparations. J. GROSSFELD and A. SIMMER (Z. Unters. Lebensm., 1928, 55, 553—559).—Comparative determinations have been carried out on commercial samples of cocoa and cocoa preparations using the scleridæ count method of Griebel and Sonntag (B., 1926, 605) and the sedimentation-crude fibre method of Grossfeld (B., 1926, 688). Generally excessive husk content is detected by both processes, but in some samples, e.g., those to which husk with few scleridæ has been added or those in which excessive fineness of the husk causes loss in sedimentation, the abnormality is only detected by one or other of the two methods. W. J. BOYD.

Cacao beans and cocoa products. V. Pigments of cacao beans and cocoa products. Ash content of cocoa paste. H. FINCKE (Z. Unters. Lebensm., 1928, 55, 559—566, 567—568).—The two pigments of cacao beans have been designated cacao-red and cacao-brown. The former is easily soluble in alcohol. It is bright red in acid solution, violet in neutral solution, and green or greenish-blue in faintly alkaline solution. Cacao-brown, to which cocoa owes its colour, is very difficultly soluble in alcohol, but easily soluble in aqueous alkaline solution. Both are tannin substances. Cacao-red occurs in the fresh seeds, in commercial fermented beans, and in the finished products, in variable quantity. There are differences in this respect characteristic of beans of different origin, but even individual beans of the same fruit differ from one another. Cacao-brown does not occur in the fresh seeds, but is gradually produced from a colourless parent substance in the various processes of fermentation, drying, roasting, and further working of the material. Cacao-red is also transformed into cacao-brown in the fermentation process and in the preparation of cocoa powder with addition of alkali. Cacao-red is produced in the living plant, probably from the same parent substance as cacao-brown. It is also produced both from cacao-brown and its parent substance by the action of alcoholic hydrochloric acid.

Determinations on material from various sources show that the ash content of the dried paste varies from 2% to 4.5%. (Cf. Adam, B., 1928, 688.)

W. J. BOYD.

Dextrins of honey and artificial honey. J. FIEHE and W. KORDATZKI (Z. Unters. Lebensm., 1928, 55, 602—608).—Dextrins were obtained from various natural and artificial honeys by precipitating proteins,

mucins, and other substances by methyl alcohol, and then precipitating the dextrins by adding alcohol and ether (cf. Lucius, B., 1926, 844). All gave Jolles' diphenylamine test for lævulose (Apoth.-Ztg., 1909, 719). Even after repeated precipitation and prolonged digestion with absolute alcohol to remove any traces of sucrose and lævulose a positive reaction for the latter substance was obtained. It is concluded that both in natural and artificial honey dextrins containing lævulose molecules occur. A method of determining the amount of the lævulose fraction approximately was found. A 1% dextrin solution is heated with two fifths of its volume of 5*N*-hydrochloric acid for 2½ hrs. on the boiling water-bath according to Lucius' method (*loc. cit.*) whereby the dextrose is unchanged but the lævulose forms among other substances hydroxy-methylfurfuraldehyde. The difference in the iodine value determined by the method of Auerbach and Bodländer (B., 1924, 567) before and after extraction of the solution with ethyl acetate is an approximate measure of the lævulose originally present. Comparison of the results with those obtained for synthetic mixtures of dextrose and lævulose indicate the presence of 20—30% of lævulose molecules in the dextrins of honey and artificial honey. W. J. BOYD.

See also A., Nov., 1192, **Electrochemistry of the proteins** (GREENBERG). 1271, **Second protein (livetin) of egg-yolk** (KAY and MARSHALL). 1272, **Freezing of milk and its fermentation** (WINTER). 1278, **Determination of tryptophan and tyrosine in proteins** (TILLMANS, HIRSCH, and STOPPEL). **Nutritive value of muscle-protein** (SEKINE). **Measuring the nutritive value of proteins** (MITCHELL). **Determination of coefficients of utilisation of foodstuffs** (HELLER, BREEDLOVE, and LIKELY). 1282, **Action of rennin on proteins in milk** (ZAYKOWSKY, FEDOROVA, and IWANKIN). 1289, **Enzyme content of germinating wheat** (PROKOPENKO). **Storage of carrots** (HASSELBRING). 1290, **Storage of mangels** (PYNE).

PATENTS.

Treatment of wheat and other cereals. J. A. HALL and W. F. C. GEORGE (B.P. 298,700, 19.7.27).—Wheat etc. is heated for 4—10 hrs. at 100—115° in a current of air saturated at about 40°, and after grinding is added to flour (¼—3 pts. per 100) to improve its baking properties. L. A. COLES.

Production of nutrient material with a high vitamin content. CHEM. FABR. DR. H. SANDER & Co. A.-G. (G.P. 448,870, 25.12.24).—Bran, yeast, etc. is extracted with phosphoric acid, the acid is neutralised with milk of lime, and the product is emulsified with cod-liver oil etc. L. A. COLES.

Irradiating milk solids with ultra-violet rays. G. M. CLARK. From DRY MILK Co. (B.P. 298,585, 11.6.27).—Milk, in dry or liquid form, is given anti-rachitic properties by agitation, e.g., by spraying, and simultaneous exposure to ultra-violet rays of such intensity and for such a period of time—which may be as little as 8 sec.—that at least 75% of the total anti-rachitic properties which can be produced in the milk

are developed before it acquires a disagreeable odour or taste.

J. S. G. THOMAS.

Food preservation. C. and J. MOHRENWEISER [C. BERTRAM] (B.P. 298,061, 14.5.28. Ger., 4.2.28).—Fresh fruit is treated either with a powder such as chalk, or with a glaze-like substance, *e.g.*, paraffin wax, neat's-foot oil, etc., to which are added small quantities of colophony, sodium benzoate, benzoic acid, salicylic acid, spiræin, and ammonium carbonate.

F. R. ENNOS.

Treatment of vegetable substances [tea leaf]. CARRIER ENGINEERING CO., LTD., and K. J. R. ROBERTSON (B.P. 297,900—1, 7.7.27).—(A) Tea leaf is subjected to a current of warm air passing through a chamber in the same direction as the leaf, in order to effect "limping" by evaporation of the moisture of the leaf and reduction of the air temperature. After being slightly damped the leaf then passes into a cool, dry atmosphere, where vigorous evaporation of its moisture occurs, causing effective "withering." (B) The leaf obtained as in (A) is conditioned or fermented by passing a counter-current of air of pre-determined temperature and humidity across its surface.

F. R. ENNOS.

Preservation of green fodder in silos, fermentation chambers, etc. I. G. FARBENIND. A.-G., Assees. of K. VIERLING (G.P. 447,905, 2.2.23).—Electrolytes capable of maintaining a degree of acidity favourable to the development of lactic acid bacteria, but not of butyric acid bacteria, *e.g.*, alkali and alkaline-earth phosphates, are added to the material.

L. A. COLES.

Increasing the usefulness of fodder and food. E. MISLIN, Assr. to J. F. and G. DAUBEK (U.S.P. 1,685,004, 18.9.28. Appl., 7.4.25).—Pentosans, after treatment whereby the yeasts and bacteria thereon are cultivated without the addition of hexoses, are allowed to act on the fodder and foodstuffs, and the mixture is then gradually heated to 50°.

F. G. CLARKE.

[Pressure chamber for] curing of raw meats, such as hams, bacons, fish, etc. M. M. HERIOT (B.P. 298,802, 2.12.27).

Pasteurising apparatus for liquids [*e.g.*, milk]. E. C. R. MARKS. From CREAMERY PACKAGE MANUF. Co. (B.P. 298,356, 23.9.27).

Yoghurt curdled milk and junket (B.P. 272,468).—See XVIII. **Theobromine from cacao etc.** (F.P. 584,781).—See XX.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Examination of extracts etc. containing fat and mucilage. J. HÜBSCHER (Süddeut. Apoth.-Ztg., 1928, 68, 205—206; Chem. Zentr., 1928, i, 2278).—For the examination of cod-liver oil and malt extracts the material is mixed with hot water, and boiled after addition of concentrated hydrochloric acid; after cooling, the liquid is shaken with ether, and the emulsion separated by addition of sodium chloride. The acid is washed out with water, and the ether evaporated. The residue, containing tragacanth mucilage and cod-

liver oil, is warmed for some time, dehydrated with sodium sulphate, and treated with ether.

A. A. ELDRIDGE.

Synthesis of camphor. Concentration of isobornyl acetate. M. TOMEO (Anal. Fis. Quim. Tecn., 1928, 26, 30—55).—Methods of producing isobornyl acetate of sufficient purity to give good yields in the synthesis of camphor are described and discussed. Criteria of purity which may be employed are, for the cruder material, the density, and, for the pure substance, the refractive index.

H. F. GILLBE.

Activity and ageing of ergot preparations. A. PRYBILL and K. MAURER (Arch. Pharm., 1928, 266, 464—479).—Fluid ergot extracts have been prepared according to directions given in various official pharmacopœias and tested for keeping qualities. All extracts deteriorate more or less rapidly, the biological activity decreasing, broadly speaking, with the alkaloid content. The extract from a dried drug which had been previously extracted with light petroleum was more stable than a similar extract from the untreated drug. Light petroleum extracts more than the fat present in the drug. Extracts made by the German and American methods have the highest alkaloid contents. Extracts rich in alkaloid are of no value so long as no means exist for stabilising such solutions, and stabilisation of the drug does not produce a stable extract.

S. COFFEY.

Analysis of fluid extract of ergot. H. KAUL (Süddeut. Apoth.-Ztg., 1928, 68, 195; Chem. Zentr., 1928, i, 2278).—For the determination of the total bases, the extract (40 g.) is shaken with magnesia (2 g.) and a mixture of equal parts of xylene and chloroform (40 c.c.). After some time 7 g. of powdered tragacanth are added, and 25 c.c. of the filtered solvent are mixed with 20 c.c. of 0.1N-hydrochloric acid and separated; the acid solution is titrated with 0.1N-potassium hydroxide solution, using methyl-orange.

A. A. ELDRIDGE.

Determination of novocaine and codeine hydrochlorides in the presence of one another. E. SCHULEK and G. VASTAGH (Arch. Pharm., 1928, 266, 452—455).—The mixed bases are extracted with chloroform from an ammoniacal solution and titrated with 0.02N-hydrochloric acid, methyl-red being used as indicator. The novocaine is then decomposed by warming with two equivalents of 0.02N-sodium hydroxide, the codeine being again extracted from the resulting solution with chloroform and titrated with 0.02N-acid under specified conditions.

S. COFFEY.

Preparation and constitution of Hydrargyrum salicylicum [mercurisalicylic acid]. E. RUPP (Arch. Pharm., 1928, 266, 504—506. Cf. A., 1926, 534; 1927, 685).—Mercurisalicylic acid is prepared by warming equimolecular proportions of sodium salicylate and mercuric sulphate on the water-bath until the product is entirely soluble in sodium hydroxide solution. The product consists of almost equal parts of *o*- and *p*-mercurisalicylic acids, the amount of the latter being accurately determined by conversion into *p*-thiocyanomercurisalicylic acid on treatment with alkali thiocyanate.

S. COFFEY.

Chemistry of mercury ointment. R. DIETZEL and J. SEDLMAYER [with E. KREMBE] (Arch. Pharm., 1928, 266, 507—517).—The determination of small quantities of mercury in the presence of large amounts of organic matter can be carried out satisfactorily by completely oxidising the latter with excess of nitric acid in a bomb tube at 150—180° and titrating the mercury with ammonium thiocyanate. Oxidation with sulphuric acid and nitric acid or hydrogen peroxide, or with potassium chlorate and hydrochloric acid, furnishes untrustworthy results. Ointments prepared from pure mutton tallow and lard may be kept for long periods without undergoing change, but if wool fat, ivory nut oil, or olive oil is incorporated, the products become rancid and considerable amounts of mercury salts are formed. The presence of mercury in the ether extract from an ointment does not necessarily mean that mercury salts of higher fatty acids are present, since ether containing ethyl peroxide dissolves metallic mercury. Mercuric stearate, palmitate, and oleate are difficult to prepare in the wet way, the purest products being obtained by treating the acid in absolute alcohol with alcoholic potash and precipitating with alcoholic mercuric chloride. Pure mercuric stearate and palmitate are obtained by heating equivalent quantities of acid and mercuric oxide to 180° and recrystallising the product from a mixture of alcohol, chloroform, and pyridine, or by dissolving the crude melt in liquid paraffin at 180° and precipitating with ether. Pure mercuric oleate was not obtained. Accurate determinations of the solubility of mercuric stearate and palmitate in various solvents are recorded. Palmitic acid affords *acid potassium palmitate*, $C_{32}H_{63}O_4K$, when titrated with alcoholic potassium hydroxide in absolute alcohol, phenolphthalein being used as indicator. S. COFFEY.

Essential oil of *Myrtus communis*, L. F. L. VODRET (Annali Chim. Appl., 1928, 18, 421—428).—The constants of (1) Sardinian myrtle oil, and (2) the same oil after distillation in a current of steam are: d_{20}^{10} 0.8961, 0.8914; α_D^{20} +18.4°, +19.2°; n_D^{20} 1.4702, 1.4708; viscosity at 20° 1.23, 1.27; solubility at 15° in 70%, 80%, and 90% alcohol 1:26, 1:11, 2:1 for each oil; acid value 1.36, 0.23; saponif. value 22.80, 36.40; acetyl saponif. value 53.10, 51.40; iodine value 201.10, 224.70; esters (as $C_{10}H_{18}O$) 7.50, 12.66%; combined alcohols 5.29, 9.94; free alcohols 25.82, 20.63% $C_{10}H_{18}O$. Neither fluoresces in Wood's light and both contain phenols and minimal traces of aldehydes, and both solidify below -12°. The presence of *d*-pinene and of two isomeric compounds, $C_{10}H_{18}O$, b.p. 170—179° and above 200°, respectively, has been detected. T. II. POPE.

Ethereal oil content of fragrant plants of the south-eastern territory. O. SOBOLEVSKI (Ber. Sartov. Naturforscherges., 1925, 1, No. 2—3, 3—38).—The yields of ethereal oil from a number of plants are recorded. CHEMICAL ABSTRACTS.

Conservation of the characteristics of emulsion of almonds. M. BRIDEL and (Mlle.) M. DESMAREST (Bull. Soc. Chim. biol., 1928, 10, 1050—1055).—See B., 1928, 729.

See also A., Nov., 1214, **New vesicant** (KIRNER).

1217, **Auto-oxidation of citronellal** (ZIMMERMANN). 1224, **Methyl salicylate glucoside of *Gauitheria procumbens*, L.** (BRIDEL and GRILLON). **Ameliaroside**, a new glucoside from *Amelanchier vulgaris*, Moench. (BRIDEL, CHARAUX, and RABATÉ). 1228, **Action of anhydrous chloral on amino-alcohols and -ethers** (FOURNEAU and BRYDOWNA). 1229, **Ureides of bromovaleric acids** (FOURNEAU and FLORENCE). 1230, **Organic compounds of arsenic** (STEINKOPF, DUDEK, and SCHMIDT). 1231, **β -Substituted alkylarsinic acids** (SCHERLIN and EPSTEIN). **Derivatives of β -aminoethyl- and γ -aminopropylarsinic acids** (GOUGH and KING). 1239, **Iodine values of sterols** (COPPING). 1240, **Oil-soluble bismuth salts** (PICON). 1253, **Indian essential oils** (RAO and SIMONSEN). 1254, **Aromadendrene** (BRIGGS and SHORT). 1265, **Strychnine phosphomolybdate** (ANTONIANI). **Alkaloids of Kurchi bark** (GHOSH and GHOSH). **Microchemistry of the alkaloids** (BECKMANN). **Arylarsenoxides and derivatives** (NEWBERY and PHILLIPS). 1266, **Organic compounds of arsenic** (STEINKOPF and DUDEK). **Mercuration of aromatic amines** (ALBERT and SCHNEIDER). 1280, **Toxicity of lead compounds** (BISCHOFF and others). 1287, **Action of heat on insulin solutions** (KROGH and HEMMINGSEN). 1288, **Extraction of ovarian hormone** (THAYER, JORDAN, and DOISY). **Irradiation of ergosterol** (WEBSTER and BOURDILLON).

Tannin of *Geranium maculatum*. PEACOCK and PEACOCK.—See XV.

PATENTS.

Production of theobromine from cacao or cocoa waste. L. M. ROUSSEAU (F.P. 584,781, 26.10.23).—The solution obtained by the exhaustive extraction of cacao etc. with calcium hydroxide solution is exactly neutralised with sulphuric acid, and ferrous sulphate is added whereby colloidal iron hydroxide is precipitated together with the theobromine and colouring matter. After drying the precipitate, theobromine is recovered from it by the usual method. L. A. COLES.

Laxative. R. PASTERNAK, ASSR. to C. PFIZER & Co. (U.S.P. 1,681,361, 21.8.28. Appl., 30.10.25).—The residues from phenolphthalein manufacture, obtained by evaporation of the alcoholic mother-liquors, contain as much as 25% of phenolphthalein, but are 20—30 times as active in laxative properties. Ether extracts the active substance, m.p. 130—200°. C. HOLLINS.

Thymol derivative (B.P. 298,393).—See XXIII.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

See A., Nov., 1197, **Technique of photochemistry** (BEESLEY and RIDYARD). **Phototropic compounds of mercury** (RAO and WATSON). 1198, **Mechanism of photosensitisation and photo-inhibition** (MUKERJI and DHAR). **Photochemical decomposition of formic acid** (HERR and NOYES). **Velocity of certain photochemical reactions** (BHATTACHARYA and DHAR; MUKERJI and DHAR; EGGERT and SCHRÖTER). 1289, **Photo-oxidation with anthraquinone** (PEILSTICKER).

Luminous flames. CHAMBERLIN and ROSE.—See II.

PATENTS.

Photographic colour screens. SOC. CIVILE POUR L'ÉTUDE DE LA PHOTOGRAPHIE ET DE LA CINÉMATOGRAPHIE EN COULEURS (B.P. 286,223, 24.2.28. Fr., 28.2.27).—To obtain even distribution of colour over the film and fastness of colours, the ordinary tinted gelatin filters used in the colour-projection of reticulated films are substituted by juxtaposed cylindrical tubes containing coloured salt solutions. The tubes may be immersed in a parallel-faced tank containing a liquid of higher refractive index than the solutions, the whole being cooled by a thermo-siphon device attached to the tank.
J. W. GLASSETT.

Providing photographic raw film with visible reproducible inscriptions. I. G. FARBENIND. A.-G. (B.P. 287,124, 24.8.27. Ger., 16.3.27).—The inscriptions are impressed on the rear side of the support for the sensitive layer by means of a solution of dye in a solvent consisting of a mixture of (a) a slowly evaporating substance which is a swelling agent for the support, thereby securing penetration of the dye, and (b) a rapidly evaporating substance which accelerates the drying of the imprint.
J. W. GLASSETT.

XXII.—EXPLOSIVES; MATCHES.

Decomposition of [propellant] powders and explosives, and the theory of stabilisers. H. MARAOUR (Chim. et Ind., 1928, 20, 610—617).—The decomposition of powders is the result of a slow hydrolysis at ordinary temperatures of the nitric esters by the autocatalytic action of the nitric acid that is formed during manufacture, *e.g.*, when the powder is being incorporated or pressed or when the solvent is being driven off. No gas is evolved during hydrolytic decomposition, which distinguishes it from a thermolytic decomposition at high temperatures. Stabilisers act by neutralising the traces of nitrogen oxides that are formed during manufacture. All stability tests that depend on measuring the gas evolved when the powder is heated have no connexion with hydrolytic decomposition, but measure the amount of stabiliser in reserve. — S. BENNING.

See also A., Nov., 1193, **Gaseous combustion at high pressures** (BONE, TOWNEND, and SCOTT). 1194, **"Explosion limits" of carbonic oxide-air mixtures** (BONE, NEWITT, and SMITH). 1229, **Nitroamino-guanidine** (PHILLIPS and WILLIAMS).

PATENT.

Mixing and spraying liquids (B.P. 297,558).—See I.

XXIII.—SANITATION; WATER PURIFICATION.

Behaviour of permutit in the softening of waters for industrial use. I. MUTTI and M. BASINI (Annali Chim. Appl., 1928, 18, 387—395).—In the reaction between sodium-permutit and calcium and magnesium salts separately, the replacement of calcium is always greater than that of magnesium, in accordance with the higher at. wt. Magnesium is replaced almost completely

from its salts by means of calcium-permutit, but only partly by sodium-permutit. From a solution containing both calcium and magnesium salts, sodium-permutit ultimately displaces a greater percentage of the magnesium than of the calcium cations, but this result appears to be brought about partly by the action on the magnesium salts of the calcium-permutit formed.

T. H. POPE.

Chemical and physical properties of water in relation to corrosion and protection against corrosion. L. W. HAASE (Gas- u. Wasserfach, 1928, 71, 1009—1016).—The theory of the corrosion of iron pipes by water is discussed in some detail. Particular attention is directed to the method of prevention of such corrosion by treatment of the water with lime, and the results of a recent successful application of the method are given.
A. B. MANNING.

PATENTS.

Indicating the termination of the period of use of breathing cartridges supplying oxygen. DEUTS. GASGLÜHLICHT-AUER-GES.M.B.H., and HANSEATISCHE APPARATEBAU GES. (B.P. 287,562, 17.3.28. Ger., 25.3.27).—A small tube placed at a suitable point in the cartridge contains a strongly tasting or smelling substance, *e.g.*, menthol, which is liberated by the fusion of a seal of low-melting metal when the temperature reaches about 60° and is discharged by a spring into the apparatus, the presence of the vapour in the inhaled air indicating that the apparatus is nearly exhausted.

L. A. COLES.

Production of a derivative of 3-[hydroxy-1-methyl-4-isopropyl]benzene [thymol]. S. EDELMAN (B.P. 298,393, 16.11.27).—Equimolecular amounts of pure, neutral, freshly-distilled trichloroacetaldehyde and pure thymol are heated at 100° to give a brown-grey, resinous, odourless product, m.p. 60°, which has disinfectant and germicidal properties.
C. HOLLINS.

Extraction of salts from aqueous solution [sea water]. D. G. ZALOCOSTAS, ASSR. to SALT PRODUCTION SYND., LTD. (U.S.P. 1,684,935, 18.9.28. Appl., 28.3.25. Austral., 10.4.24).—Sea water is evaporated at high temperature until the concentration is just below that at which calcium sulphate is precipitated, the brine is transferred to another closed chamber and is evaporated at 38° under reduced pressure until the calcium sulphate is precipitated as mud, and the evaporation is continued in another evaporator to precipitate sodium chloride; finally the residual liquor is removed. — W. G. CAREY.

Water still. E. V. OLIVER (U.S.P. 1,686,418, 2.10.28. Appl., 29.12.23. Renewed 14.6.28).—The conical condensing surface of a water still is provided on the cooling-water side at the upper part with a baffle causing circulation of the water.

B. M. VENABLES.

[Steam] sterilising process. A. LESEURRE (B.P. 286,239, 9.7.27. Fr., 1.3.27).

Device for softening or purifying water. G. W. CONDUIT (B.P. 297,943, 18.2.28).

Sterilising apparatus (B.P. 297,589).—See I.

BRITISH CHEMICAL ABSTRACTS

B.—APPLIED CHEMISTRY

DEC. 21, 1928.



I.—GENERAL; PLANT; MACHINERY.

Milling material. Distribution of particle size in the ground product. A. H. M. ANDREASEN (Kolloidchem. Beih., 1928, 27, 349—458).—Mainly a theoretical and mathematical investigation of the possible methods of characterising a finely-ground substance. It is considered that a single numerical value, representing, for example, the increase in surface per kg. of substance, does not suffice to characterise the material, and it is essential to consider the distribution of the various sizes of particles. This may be investigated by sieving, by flotation, or by sedimentation. Several definitions of the new ideas involved are given, and the relations between these are worked out mathematically. A number of mills are described, and with their aid the characteristics of the new terms have been investigated experimentally on felspar, flint, glass, brick, barytes, iron glance, and quartz sand. It is concluded that no general relation exists for the distribution of particle size in a milled material, the results varying with the substance examined.

E. S. HEDGES.

Measurement of high temperatures by means of a tungsten-molybdenum thermocouple. D. BINNIE (J. Roy. Tech. Coll. Met. Club, Glasgow, 1927—8, [6], 35—36, 42).—A tungsten-molybdenum thermocouple may be used for temperature measurements up to 2000° provided that oxidation is avoided by passing a current of hydrogen through the furnace or through the protecting tube of the couple. The thermo-electric curve is almost a parabola with the equation $y = 0.151x^{2.125}$, the neutral temperature occurs at 618° when the *E.M.F.* of the junction is 1.36 millivolts, and the reversal point at 1218° using a cold junction at 18°. Above the reversal point tungsten is the electropositive metal. The tungsten wire should not exceed 0.4 mm. in diam. nor the molybdenum wire 0.64 mm.

A. R. POWELL.

PATENTS.

Maintaining an even temperature in chambers in which exothermic reactions are carried out. SOC. CHIM. DE LA GRANDE PAROISSE, AZOTE & PROD. CHIM. (F.P. 626,501, 1.4.26).—Heat is transferred from the hot end of the catalyst to the cooler end by a suitable heat-interchange system or by passing the gases, before they enter the reaction chamber, over the hotter end of the chamber to preheat them before they pass to the cooler part of the catalyst.

A. R. POWELL.

Mercury vapour generators. BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF B. L. NEWKIRK (B.P. 295,021, 24.7.28. U.S., 4.8.27).—A mercury vapour generator for removing the heat from the walls of furnaces comprises a series of tubes 10 ft. long and 0.4 in. internal

diam. communicating at their lower ends with a supply pipe and heater which generates the mercury vapour in such a way that the vapour is charged with 2—3% of its volume of finely-divided globules of liquid mercury. This vapour rises through a helical spiral inside the tubes and acts as a more efficient heat-interchange substance than mercury vapour free from liquid.

A. R. POWELL.

Separation of substances of varying sp. gr. A. K. and A. K. DOBREV and D. K. IONOMOV (F.P. 622,309, 30.9.26).—Constituents of different sp. gr. may be separated from mineral mixtures by allowing the mixture to flow down a sloping trough up which a current of liquid is pumped, whereby the lighter particles are carried upwards while the heavier particles sink to the bottom of the trough.

A. R. POWELL.

Apparatus for ascertaining the characteristics of flowing liquids. V. HENNY and J. D. SEGUY, ASSRS. to UNIVERSAL OIL PRODUCTS CO. (U.S.P. 1,688,811, 23.10.28. Appl., 5.7.23. Renewed 2.7.28).—The liquid flows into a constant-level container communicating with a second container of the same height in which a stagnant sample of the liquid collects.

L. A. COLES.

Apparatus for mechanical treatment of a mixture of gas and liquid. W. L. J. SPOOR (F.P. 624,383, 9.11.26. Holl., 10.11.25).—The gas mixture is passed through a centrifugal or screw valve in such a way that the valve opposes the flow of gas, whereby the suspended particles of liquid are deposited on the walls of the valve by the centrifugal force.

A. R. POWELL.

Purification of gases. W. NEU (F.P. 624,542, 13.11.26. Ger., 14.11.25).—The gas stream is first given a high velocity and then the rate of flow is rapidly retarded, whereby the solid impurities are deposited or carried along in the original direction of flow whilst the purified gas is taken out of the circuit through side tubes.

A. R. POWELL.

Rotary kiln and furnace. J. H. BENTLEY (U.S.P. 1,690,048, 30.10.28. Appl., 9.9.26. U.K., 8.1.26).—See B.P. 263,630; B., 1927, 175.

Heat exchanger. C. H. POTTS, ASSR. to VICKERS, LTD. (U.S.P. 1,690,501, 6.11.28. Appl., 11.1.28. U.K., 8.10.26).—See B.P. 285,524; B., 1928, 506.

Minute disintegration of substances. Disintegrating device. Production of suspensions. F. J. E. CHINA (U.S.P. 1,690,667—9, 6.11.28. Appl., [A] 10.12.23, [B] 12.3.26, [C] 25.2.28. U.K., [A—C] 8.3.22).—See B.P. 193,693; B., 1923, 386 A.

Apparatus for mixing liquids with solid or semi-solid substances or with other liquids. N. BENDIXEN, ASSR. to J. G. Y. D. MORGAN (U.S.P. 1,689,103, 23.10.28.

Appl., 27.11.26. U.K., 25.5.26).—See B.P. 266,950; B., 1927, 320.

Separation of liquids. H. S. HELE-SHAW and A. BEALE (U.S.P. 1,690,537, 6.11.28. Appl., 19.7.26. U.K., 22.7.25).—See B.P. 260,045; B., 1927, 1.

Drum for centrifugal separating apparatus. K. J. SVENSSON and K. A. P. NORLING (U.S.P. 1,688,838, 23.10.28. Appl., 11.12.26. Swed., 29.12.25).—See B.P. 263,443; B., 1927, 128.

Process for [rotary] filtering elements. II. A. VALLEZ (U.S.P. 1,691,001, 6.11.28. Appl., 19.5.22).—See B.P. 201,822; B., 1923, 961 A.

Gas burners for heating furnaces etc. E. E. LUCAS (B.P. 299,125, 12.4.27).

Refrigerating systems. U. R. and H. R. LORANGER (B.P. 298,498, 9.5.27).

[Whisking] machines for mixing, aerating, and like treatment of various materials [e.g., cakes, confectionery]. BAKER PERKINS, LTD., L. S. HARBER, and J. E. POINTON (B.P. 298,723, 9.8.27).

Gas cartridges for fire extinguishers. G. SCHWORETZKY, and EXCELSIOR FEUERLÖSCHGERÄTE A.-G. (B.P. 299,096, 22.7.27).

Gas purifier (F.P. 623,660).—See II. Purification of gases (B.P. 298,726).—See VII.

II.—FUEL; GAS; TAR; MINERAL OILS.

X-Ray examination of coal sections. C. N. KEMP (Proc. Roy. Soc. Edin., 1928, 48, 167—176).—The technique of preparing coal sections for X-ray examination is described in detail and illustrated photographically. The block of coal is placed in a wooden box and embedded in plaster of Paris. The whole is then cut into sections by means of a band-saw or, if necessary, a reciprocating power-saw. If the cut surface is chipped it may require subsequent milling or grinding. Sections $\frac{1}{2}$ in. in thickness appear to be the most suitable for giving details of structure and ash distribution. The interpretation of the photographs is briefly discussed.

A. B. MANNING.

Origin of fusain. H. BODE (Mitt. Abt. Gesteins-, Kohle-, u. Salz-Untersuch., 1927, 12—22; Fuel, 1928, 7, 487—492).—The theories of Stach and of Petrascheck on the origin of fusain are adversely criticised. A study of the occurrence of fusain in coal balls and in the sphärosiderites of brown coal proves that wood charcoal was already present in the primeval peat in the same form in which it is now found as a constituent of bituminous and brown coal, and supports the forest-fire theory of its origin.

A. B. MANNING.

Swelling of coal. G. LAMBRIS (Brennstoff-Chem., 1928, 9, 341—346).—Existing tests for the swelling power of coals, such as those of Muck, Kronig, Korten, and others, are all open to the objection that they do not allow the maximum possible expansion, and the results of different investigators cannot be compared owing to lack of uniform conditions. The formation of a film of coke on the outside of the test coal will suppress expansion as it becomes rigid, and swelling will stop when the coke layer is complete; it will reach a maximum only

when the coal particles remain movable for some time after they are heated. This is achieved by putting a layer of kieselguhr at the bottom of a crucible, with powdered coal above, and heating at 900° without a cover; the coke film quickly formed on the top acts as an insulator, but does not prevent expansion as the coal is heated further only from the sides. Compared with cokes produced by other methods, the volume per unit volume of coal is more than twice as great, the apparent density is very low, the porosity high, and the yield lower. Other materials which might be used to form the layer in the crucible are decreasingly useful in the order of their increasing heat conductivities.

R. H. GRIFFITH.

Comparative action of bromine on cellulose, lignin, wood, and coals. W. FUCHS (Brennstoff-Chem., 1928, 9, 348—350).—When wood is treated with bromine, the cellulose is unaffected but the lignin reacts slowly and a definite proportion of the halogen can then be displaced by the action of aqueous acetate solution. The substance (2 g.) to be examined is suspended in 25 c.c. of carbon tetrachloride, and 25 c.c. of a 4% solution of bromine in the same solvent are added, the whole is boiled for 20 min., filtered, and washed. Bromine is determined by heating with a soda-potash mixture, and the removable bromine by boiling with 50 times its weight of 5% acetate solution; in the case of coal, calcium acetate is used. Air-dried wood takes up about 10% of bromine, most of which is removable as hydrogen bromide, and lignin reacts even when quite dry. Coals are extracted with benzene-alcohol mixtures before treatment, and are found to react readily; after removal of part of the bromine with acetate solution the residue will add on more halogen than it took up originally.

R. H. GRIFFITH.

Absorption of pyridine vapour by Upper Silesian coals. W. SWIENTOSLAWSKI and M. CHORAŹY (Przemysł Chem., 1928, 12, 526—534).—Vitrain on being kept in a desiccator containing pyridine will absorb 70% of its dry, ash-free weight of pyridine vapour, durain 54%, and fusain 12%. Under the same conditions benzene, aniline, dimethylaniline, and quinoline vapours are absorbed only to a negligible extent. A pyridine extract of coking coal will absorb over 100% of its weight of pyridine vapour. A convenient basis is thus afforded for determining the coking qualities of a sample of coal.

R. TRUSZKOWSKI.

Coke formation. II. R. A. MOTT and T. SHIMMURA (Fuel, 1928, 7, 472—486; cf. B., 1927, 353).—A number of Yorkshire and Durham coals have been heated, both in the lump and in the powdered form, to 900°, at a uniform rate of either 1° or 5° per min. With the slower rate of heating the swelling was roughly proportional to the carbon content of the coals, which varied from 82 to 85%. No correlation could be observed between the degree of swelling and the rate of gas evolution during the plastic period. The influence of the rate of tar evolution on the coking process was studied by comparing the cokes produced from coals which had been preheated to various temperatures (390—450°), before and after extraction of the preheated coal with ether. It is suggested that the first stage of coke forma-

tion from crushed coal is the liquation of tar to the surfaces of the particles until these are completely wetted, and the second stage is the loss of identity of the coal particles with the formation of pore structure due to swelling.

A. B. MANNING.

Oxidation of hydrocarbons. DUMANOIS and MONDAIN-MONVAL (Compt. rend., 1928, **187**, 892—894).—The temperature-pressure curves of pentane-air mixtures show that for less than 5–10% of the theoretical amount of pentane required for combustion no ignition occurs below 300°, though at 250° there is a slight increase in pressure (cf. Lewis, A., 1927, 851). For larger quantities of pentane the graph is linear but shows a change of direction, corresponding with a relative increase in pressure, at 120°, whilst 10–15° below the spontaneous ignition point, at a temperature which decreases as the proportion of pentane increases, the pressure mounts very rapidly. Pentane-nitrogen mixtures give a steeper line with no angular point. In the presence of 0.001% of lead tetraethyl more than 10% of the theoretical amount of pentane is required for spontaneous combustion below 300°, the change in direction is less marked, and the final rise in pressure occurs at a slightly higher temperature. J. GRANT.

Excess air in gas heating. H. MÜLLER (Gas- u. Wasserfach, 1928, **71**, 971—975, 995—1000, 1017—1020, 1042—1046).—The theory of combustion as applied to the domestic gas fire has been studied in detail. In addition, an experimental study has been made of the relationship between the rates of supply of gas and air to the fire, the composition and temperature of the flue gases, and the amount of water which condenses in the flue. An air supply 40% in excess of the theoretical is considered to be the practical minimum. Owing to faulty installations much higher values are common. To attain complete combustion with only 40% excess air, correctly designed burners are essential. To limit the necessary heating surface the flue-gas temperature should not be too low; it should also be possible to lower the dew point of the flue gas by the addition of secondary air without unduly lowering the temperature of the mixed gases. The volume of flue gas produced per unit volume of combustible gas supplied is roughly inversely proportional to the hourly gas consumption. It therefore appears impossible to avoid high values for the excess air with very low rates of gas consumption. Satisfactory control of the excess air at normal rates of gas consumption can be attained by the use of suitable dampers in the flue. A constant value for the excess air with a varying gas consumption has been attained in the past only by subdivision of the heating units. It appears possible that such control may be achieved to some extent by regulation of the primary air to the burners.

A. B. MANNING.

Primary tar oils. R. WEISSGERBER (Ber., 1928, **61**, 2111—2119).—Fractions of the oil, b.p. 187—207°, are shaken with 2% of concentrated sulphuric acid, whereby easily resinifiable indenenes and coumarones are removed, leaving a portion, b.p. 193—201°, which is treated with 90% sulphuric acid at 15—20°. The olefines are converted thereby into alkyl hydrogen sulphates, which remain in the oil, from which they are

removed by water and then converted into their sodium salts. The acid layer retains ketonic oils (see later). The sodium salts are decomposed with steam, thus yielding methylnonylcarbinol, further identified by conversion into the oxime of methyl nonyl ketone, m.p. 45—46°. The presence of undecylene in the oil is thus established. Its amount cannot be deduced from these experiments, but the occurrence of olefines in the oil accounts for the difficulty of purifying the latter by the customary technical methods. The oil fraction (see above) contains normal alkyl sulphates in such quantity that it cannot be distilled under reduced pressure. Treatment of it with steam at 150° yields a distillate with the properties of a technically pure hydrocarbon residue and a large proportion of dark yellow resin from which sulphuric acid is removed by aniline at 170° in small amount; the production of the resin is mainly due to polymerisation of olefines. The oil dissolved by the 90% sulphuric acid (see above) is distilled with steam, thereby leaving a small amount of resin. About one third of the distillate consists of ketones which are removed as hydrazones, leaving a mixture of nitriles (from the hydrolytic product of which *o*-toluic acid is isolated) and polysubstituted thiophenes, chiefly tetramethylthiophen. The latter compounds do not afford additive compounds with mercuric chloride, lose sulphur as sulphuric acid when oxidised, and give uninviting products with halogens. Their nature is established by partial dealkylation by passage through a tinned iron tube at 650—675°, whereby thiophen, thiotolen, 2:3-dimethyl- and trimethyl-thiophen are produced. 2:4-Dimethylthiophen at 675—700° similarly yields thiophen and thiotolen. 2:5-Dimethylthiophen is converted by successive acetylation and reduction according to Clemmensen into 2:5-dimethyl-3-ethylthiophen, which, by repetition of the processes, affords 2:5-dimethyl-3:4-diethylthiophen, b.p. 214—217°, d^{15}_4 0.9573; the compound dissolves unchanged in 80% sulphuric acid. Comparison of the behaviour of the primary tar-oil fraction with that of a coke-oven tar fraction of similar b.p. shows that the exposure to the higher temperature greatly diminishes the amounts of olefines, ketones, and homologous thiophenes partly by gasification and partly by dealkylation. The apparent appreciable increase of coumarones, indenenes, and nitriles is due to their remarkable thermostability at 900—1000°. In the chemical character of their groups the tars are identical. The quantitative distribution of these groups and the predominance of homologues in primary tar impart distinctive character to the products. H. WREN.

Ammonia recovery from steamed vertical-retort gas. C. SINGLETON and J. E. STANDER (Gas J., 1928, **184**, 405—407).—In vertical retorts where the yield of ammoniacal liquor per ton of coal depends largely on the amount of steam added to the retorts, the recovery of the ammonia in the highest concentration possible is of great importance. At Stockport gas from 48 retorts with a capacity of 7 tons per day each passes through water-cooled condensers, exhausters, washers, and tar extractors, and finally to an acid washer where the solution is worked up to about 35—38% ammonium sulphate containing 4—5% of free acid. A series of tests carried out showed that weak liquors free from

tar could be advantageously used to wash the gas, and that the introduction of the acid-catch washer increased the strength of the virgin liquor by $\frac{3}{4}$ oz.; no trouble was experienced with entrained acid. Figures were also obtained showing the decrease in the total oxygen absorption of the spent-liquor effluent and in the total volume of liquor to be handled. R. H. GRIFFITH.

Evaluation of used transformer and switch oils for further use. K. TYPKE (Brennstoff-Chem., 1928, 9, 346—348).—Physical and chemical tests can be applied in examination of oils which have been used; for cases where no great heating occurs changes will not be large. The sp. gr. at 20° should not be above 0.895, and in a light oil separation of water and suspended solids will take place more readily. Viscosity may be between 10° and 11° Engler, the solidifying point should be below -40°, and the dielectric strength not less than 80 kv. per cm. Among chemical tests, the ash content, which is related to the amount of soap present, should be below 0.2%, the acid value 1.0, and the saponif. value 4.0. Examination with caustic soda or sulphuric acid does not give any important information, but miscibility with other oils should be considered; this is determined by mixing, keeping over-night, and measuring the sludge obtained on centrifuging.

R. H. GRIFFITH.

Artificial ageing of turbine oils. R. SCHMIDT (Z. angew. Chem., 1928, 41, 1197—1201).—In order to predict the stability of turbine oils under technical conditions it is essential that any artificial ageing (oxidation) experiments be conducted in the presence of metallic catalysts, as the presence of metals is an important factor revealing differences of behaviour of apparently similar oils. The following test is advocated: 125 g. of oil are oxidised by a slow stream of oxygen (2 bubbles per sec.) in a 250 c.c. flask immersed in a boiling water-bath; the catalyst is introduced in the form of a piece of copper foil (40 × 50 × 0.5 mm.) superficially oxidised and bent to the form of a cylinder. After 70 hrs. oxidation the oil is tested for acidity and tarry and asphaltic matter. E. LEWKOWITSCH.

Separations of emulsions. PAVLIKOVSKI.—See XI.
Lamp-blacks for rubber mixtures. KIRCHHOFF.—See XIV.

PATENTS.

Retorts for distilling coal, shale, and other fragmentary solid material. AMER. HYDROCARBON Co., Inc. (B.P. 270,662, 5.4.27. U.S., 4.5.26).—A horizontal or slightly inclined annular retort for the low-temperature distillation of coal etc. has both the inner and outer walls rotatable, either together or independently. The outer wall is conical, so that the cross-section of the annular chamber increases towards the discharge end. The walls are provided with inwardly projecting longitudinal fins. Both walls are heated and are jacketed, the interspaces containing a suitable heat-distributing material, e.g., molten lead. The inner wall is movable longitudinally in such a way that the fins attached to it may be brought into contact with the outer wall for detaching any material adhering thereto.

A. B. MANNING.

Low-temperature distillation apparatus. H. G. LYKKEN (U.S.P. 1,685,496, 25.9.28. Appl., 23.5.24).—The material to be distilled moves along a heated pan of annular shape provided with inlet and outlet ports for the material.

C. O. HARVEY.

Utilisation of heat contained in coke discharged from coke ovens, gas retorts, etc. N. V. SILICA EN OVENBOUW MIJ., and W. HIBY (B.P. 298,547, 30.6.27).—The sensible heat of the coke is utilised for the distillation, at comparatively low temperatures, of coal, shale, etc., by charging the two materials together into a chamber to which is connected condensing plant for the recovery of the volatile carbonisation products. The mixed coke and semi-coke may be used as a whole, e.g., as a domestic fuel, or may be separated by hand or by screening. The heat remaining in the coke mixture may be used to dry a further quantity of moist coal by feeding the latter into the lower part of the chamber, and subsequently separating it again by screening.

A. B. MANNING.

Recovery of sulphur [from furnace gases]. W. H. HOWARD (U.S.P. 1,685,231, 25.9.28. Appl., 20.8.27).—The gases are scrubbed with water, the resulting acid wash-water is aerated, and the mixture of air and sulphur dioxide is passed over incandescent carbonaceous material.

F. G. CLARKE.

Flotation processes for the treatment of coal, ores, etc. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 298,736, 19.8.27).—The products obtained by oxidising montan wax or paraffin wax at not above 200° and completely or partially neutralising with ammonia or other bases are employed as flotation agents.

H. S. GARLICK.

Production of adsorption agents from carbonaceous materials. O. SCHÖBER (B.P. 298,546, 9.6.27).—Highly active carbons, characterised by containing at least 10% of free carbon and over 20% of acid-soluble ash, are produced by treating carbonaceous material at 500—800° with activating gases in which the oxygen partial pressure lies between 8 and 115 mm., until the loss of weight amounts to more than 40%. The material may be given a short after-treatment at about 1000° in an oxygen-free atmosphere. A form of apparatus is used in which the waste gases from the activation and from the heating chamber are used to preheat the raw material and the activating gases or vapours. In order to free the product from sulphur and phosphorus the hot material may be given a limited acid treatment and subsequently washed, or iron in some suitable form may be added to the raw material and the product subjected to magnetic separation in a field of small intensity. Further treatment in magnetic fields of greater intensity may be used to separate the product into fractions of varying activity and ash content.

A. B. MANNING.

Gas purifier. G. VIGREUX, and SOC. ANON. DES ANC. ÉTABL. GÉPÉA (F.P. 623,660, 25.10.26).—A gas-purification apparatus comprises a metal container divided into two sections through which the gas passes in succession. The first section is filled with a liquid solvent and is provided with a series of perforated

shelves through which the gas stream must pass, whereby it is continually broken up into small bubbles. The second section is packed with a suitable dry purifying agent, which acts as a filter for solid and liquid particles.

A. R. POWELL.

Distillation of coal tar. C. WESSEL (B.P. 299,290, 24.7.28. Addn. to B.P. 273,675; B., 1928, 778).—Tar is heated to 180° to drive off water and light oils. Heating is then reduced so that when slightly superheated steam is driven into the tar the temperature does not rise above 215°, and practically oil-free naphthalene is recovered. For continuous working, the tar may be preheated to 180° in a separate container, passed to the boiler where it is steam-treated, and continuously passed out naphthalene-free.

H. S. GARLICK.

Bituminous compositions for use as supports exposed to high pressures. R. STEIN (B.P. 299,208, 8.8.27).—Epure (natural Trinidad asphalt freed from earthy impurities), goudron (a distillation product of Trinidad asphalt), and pitch, in the proportions 1 : 2 : 2, are raised to the b.p. and mixed with 1 pt. of asbestos fibres. The composition is preferably used in conjunction with one or more layers of wire.

H. S. GARLICK.

Purification of montan wax. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299,133, 20.8.27).—An aqueous solution or suspension of montan wax is subjected to the anodic action of an electric current in the presence of electrolytes and oxygen transferers, such as water-soluble compounds of chromium, cerium, vanadium, and manganese, and, if desired, an organic solvent.

H. S. GARLICK.

Oxidising paraffins, waxes, etc. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 298,704, 22.7.27).—The employment of an inorganic catalyst associated with an organic nitrogenous base (*e.g.*, a manganese soap and hexamethylenetetramine) considerably accelerates the oxidation of paraffin hydrocarbons by gases containing oxygen.

H. S. GARLICK.

Continuous transformation into light hydrocarbons of the heavy hydrocarbons from petrols and the like. H. CARROLL (B.P. 284,345, 28.1.28. Fr., 28.1.27).—Crude oil is distilled in a tubular boiler connected to a thermostatically controlled dephlegmator from which the products remaining liquid pass to a cracking apparatus. Cracking is carried out in an atmosphere rich in free hydrogen, and is obtained by causing the oil to flow in thin layers over the surfaces of metallic baths arranged in the form of steps, successively larger or wider and raised to a sufficiently higher temperature to cause the liquid to assume the spheroidal state, in order to obtain a superheating of the gaseous phase without corresponding superheating of the liquid phase.

H. S. GARLICK.

Converting or cracking heavy oils into lighter oils. E. C. R. MARKS. From PETROLEUM PROCESS CO. (B.P. 299,347, 13.6.27).—In a treating chamber preheated oil is sprayed through nozzles against baffles and falls through gratings counter-current to superheated steam. The mixed vapours are reheated and pass to a chamber similar to the first where fresh oil, admitted at

the top, flows counter-current to the vapours admitted at the bottom. Reheating of the vapours and contact with fresh oil may be repeatedly carried out before passing them to the coolers and condensers.

H. S. GARLICK.

Conversion of hydrocarbon oils into lighter oils. C. ARNOLD. From STANDARD DEVELOPMENT CO. (B.P. 299,683, 22.9.27).—In the process of cracking oil by passing it through a heated coil and digestion drums, so arranged that one drum may be used while another drum is being cleaned, the drum to be brought into use is filled with heated oil previous to diverting from the drum in use the oil from the heater coil. Preferably a portion of the oil heated in the coil is passed into the digestion drum prior to the diversion of the main stream of heated oil thereto.

H. S. GARLICK.

Manufacture of viscous [mineral or tar] oils. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299,086, 21.7.27, 19.9.27, and 18.4.28).—Oils of high viscosity and flash point are produced by passing gases rich in lower olefines into hydrocarbon mixtures which are liquid at ordinary temperatures, in the presence of catalysts of the Friedel-Crafts type and at temperatures up to 200°. As starting materials olefines obtained by subjecting tar oils or mineral oils, or higher fractions of these, to a cracking process may be used.

H. S. GARLICK.

Reconditioning of mineral oils. C. J. RODMAN and M. HECHT (U.S.P. 1,685,681, 25.9.28. Appl., 11.11.25).—Electrical insulating mineral oils are reconditioned by evacuation, treatment with an evacuated adsorbent, and filtration, the whole operation being conducted out of contact with atmospheric gases.

C. O. HARVEY.

Refining or fractionating of oils. A.-G. F. KOHLENSÄURE-IND., and E. B. AUERBACH (B.P. 285,064, 23.8.27. Ger., 12.2.27. Addn. to B.P. 277,946; B., 1928, 843).—The treatment of oils as previously described is extended to mixtures of mineral oils with fatty oils, fats, terpenes, tar oils, resin oils, ketone oils, and essential oils, or to oils containing no mineral oil.

H. S. GARLICK.

Manufacture of [rubber-like] hydrocarbons of high mol. wt. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 298,584, 9.6.27).—Products, consisting substantially of 4-carbon olefines having at least two, preferably conjugated, double linkings, obtained from the destructive hydrogenation of coal, oils, etc., are polymerised to rubber-like compounds by treatment with metallic sodium, stannic chloride, carbon dioxide, etc., or by prolonged heating. Examples of suitable materials are the fraction, b.p. about 100°, from hydrogenated mineral oil; isoprene from a hydrogenated coal fraction, b.p. 30–40°, which has been passed over chromium oxide at 500°, converted into amyl chloride by hydrochloric acid, passed at reduced pressure over barium chloride at 400° to give β -isoamylene, which is chlorinated and passed again over hot barium chloride; and butadiene prepared by cracking a hydrogenated coal fraction of b.p. 65–75°.

C. HOLLINS.

Active carbo-mineral product [mineralised charcoal]. E. URBAIN, ASSR. to URBAIN CORP. (U.S.P.

1,689,647, 30.10.28. Appl., 5.5.26. Fr., 25.6.25).—See B.P. 254,262; B., 1926, 1003.

Treating aqueous emulsions of tar. B. JOHNSON (U.S.P. 1,689,728, 30.10.28. Appl., 6.3.25. Swed., 12.3.24).—See B.P. 284,401; B., 1928, 220.

Improving the properties of montan wax. W. PUNGS and M. JAHRSTORFER, Assrs. to I. G. FARBENIND. A.-G. (U.S.P. 1,690,876, 6.11.28. Appl., 10.9.27. Ger., 13.9.26).—See B.P. 289,621; B., 1928, 473.

Cracking of liquid hydrocarbons. O. D. LUCAS and E. L. LOMAN, Assrs. to V. L. OIL PROCESSES, LTD. (U.S.P. 1,690,416, 6.11.28. Appl., 23.7.25. U.K., 9.12.24).—See B.P. 248,115; B., 1926, 396.

[Rotary] washing apparatus for coal or the like. A. P. DENBY (B.P. 299,148, 7.9.27).

Apparatus for cooling of coke. ILLINGWORTH CARBONIZATION Co., LTD., and S. R. ILLINGWORTH (B.P. 298,658, 15.6.27. Addn. to B.P. 248,078; B., 1926, 395).

Scavenging [and removing solid] combustion products in internal-combustion engines. J. Y. JOHNSON. From I. G. FARBENIND A.-G. (B.P. 299,139, 27.8.27).

Apparatus for burning liquid fuel. J. J. KERMODE (B.P. 299,486, 21.6. and 9.7.27).

Hydrocarbon oil burner. W. TEALE and H. F. McLAUTHLIN (B.P. 299,348, 23.6.27).

III.—ORGANIC INTERMEDIATES.

Partial oxidation of methane and ethane in the presence of catalysts. T. E. LAYNG and R. SOUKUP (Ind. Eng. Chem., 1928, 20, 1052—1055).—The catalytic partial oxidation of mixtures of methane and ethane has been studied at atmospheric pressure by the dynamic method within the temperature range 100—700°. Some experiments have also been made with natural gas under similar conditions. For the production of alcoholic or aldehydic intermediates, solid catalysts were unsatisfactory from the point of view of the low yield of the required end-products. The addition of 0.3—5% of nitrogen tetroxide, however, exerted considerable promoting action on the oxidation of the hydrocarbon-oxygen mixtures, and yields of 15—30% by vol. of the oxygenated derivatives were obtained. A temperature of 600° and a heating time of 0.5 sec. were found necessary. The effect of using auxiliary catalysts, such as activated charcoal or platinum oxide, in conjunction with nitrogen tetroxide resulted in an increased yield of hydrogen, oxides of carbon, and steam, but a decrease in the amount of oxygenated compound formed. Methyl nitrite also acts as a catalyst probably owing to its thermal decomposition into nitric oxide or nitrogen tetroxide and a carbon-hydrogen-oxygen residue, the former only having a catalytic action. G. CLAXTON.

Synthesis of methanol [methyl alcohol]. E. AUDIBERT and A. RAINEAU (Ind. Eng. Chem., 1928, 20, 1105—1110).—The results of the action of various substances in catalysing the reaction $\text{CO} + 2\text{H}_2 = \text{CH}_3\cdot\text{OH} + 27,000 \text{ g.-cal.}$ are given.

The following oxides: alumina, silica, molybdenum oxide (Mo_2O_3), vanadium oxide (V_2O_3), blue tungsten oxide (W_2O_3), thoria, titania, magnesia, lime, barium oxide, and strontia were inactive. Cerium oxide (Ce_2O_3), uranium oxide (UO_2), beryllia, and zirconia gave less than 2% of methyl alcohol at 150 atm. Manganous oxide, chromium sesquioxide, and zinc oxide, prepared by precipitation from the corresponding nitrates, converted 2—17.5% of carbon monoxide into methyl alcohol, the highest conversion being for zinc oxide at 350°. Metallic copper was found to be a good catalyst if prepared at a low temperature by reduction of copper oxides obtained by precipitation of the nitrate or by the thermal decomposition of copper salts of organic acids. Copper catalysts prepared from the fused oxide or from copper oxide obtained by precipitation of solutions of the chloride or sulphate were inactive. Nickel and iron catalysts favoured the production of carbon dioxide. Single-component catalysts were sensitive to heat, high temperatures impairing their activity. Mixed catalysts were more effective than single-component catalysts. A mixture of cerium oxide and copper was very effective, and the activity reached a maximum at a Ce:Cu ratio of 30:100 with a 92% conversion of carbon monoxide. A mixture of beryllia and copper with a Be:Cu ratio of 6:100 was almost as good, and showed the greatest heat resistance of the whole series studied. G. CLAXTON.

Values obtained in synthesis of methanol [methyl alcohol]. A. C. FIELDNER and R. L. BROWN (Ind. Eng. Chem., 1928, 20, 1110—1112; cf. Brown and Galloway, B., 1928, 780).—A comparison of the values obtained at 400° by the authors with those obtained by Audibert and Raineau (preceding abstract) and by Lewis and Frolich (B., 1928, 359) has been made. Using a development of the Nernst approximation formula, K_p has been calculated from the results of different workers, assuming no departure from the perfect gas laws. The values of K_p obtained are of the same order and vary as might be expected from the activities of the catalyst employed and the relative space velocities used. Taking into account deviations from the perfect gas laws, the fugacities (effective pressures) and the partial fugacities of the components have been calculated from the results of the three sets of investigators. From the partial fugacities a new set of K values, designated K_f , is calculated; the values, as for K_p , are of the same magnitude. Space-time yields are also discussed. G. CLAXTON.

Iron oxide colours. BUDNIKOV.—See XIII.

PATENTS.

Transformation into water-soluble form of organic substances which are per se insoluble in water. A. L. MOND. From I. G. FARBENIND A.-G. (B.P. 298,823, 11.1.28).—Insoluble substances, such as tetrachloroethylene, eucalyptus oil, menthol, etc., give concentrated "solutions" in solvent mixtures consisting of Turkey-red oil (40 pts.), potassium ricinoleate (10 pts.), paraffin oil (5—10 pts.), an alkali salt of an alkylnaphthalenesulphonic acid (3—5 pts.), and water (10—20 pts.), especially in presence of a little acid. An 82% solution of tetrachloroethylene, or 4% of eucalyptus oil, in this

mixture may be diluted without the precipitation which occurs in the absence of paraffin. C. HOLLINS.

Chlorination of saturated hydrocarbons with a graphite catalyst. VER. F. CHEM. U. MET. PROD. (F.P. 605,950, 12.11.25).—Chlorine and hydrocarbon vapour are passed separately into a mixing vessel filled with graphite from which the mixture passes to the reaction chamber. Alternatively, the mixed gases are passed directly through a hot chamber packed with a graphite catalyst. The reaction mixture is strongly cooled to condense the greater part of the chlorinated hydrocarbon, and the remainder is separated by washing out the hydrogen chloride in a stream of water.

A. R. POWELL.

Recovery of concentrated from dilute acetic acid. H. SUIDA (B.P. 280,501, 22.7.27. Austr., 9.11.26).—In the process of B.P. 230,447 (B., 1925, 827) aliphatic esters of the hydroxylated products are substituted for the latter as solvents, e.g., acetates of glycol, glycerol, etc., amyl acetate, amyl butyrate, hexyl acetate. The acetates may be produced *in situ*. C. HOLLINS.

Manufacture of acetaldehyde from acetylene. J. Y. JOHNSON. From I. G. FARBENIND. A.-G. (B.P. 299,234, 2.1.28).—To prevent corrosion by mercuric sulphate solution the reaction vessel is made of or lined with a chromium-nickel-molybdenum steel, e.g., 70% Fe, 19–20% Cr, 7–8% Ni, and 2–3% Mo.

C. HOLLINS.

Manufacture of aliphatic [acetic] anhydrides. BRIT. CELANESE, LTD., H. DREYFUS, and C. I. HANEY (B.P. 298,667, 14.7.27).—Acetic anhydride is extracted from vapours produced by the pyrogenic decomposition of acetic acid at 600–1000° by means of a light petroleum and ether mixture. The vapours are mixed with solvent vapour and passed into cooled solvent, water being drawn off from the bottom of the extracting vessel and acetic anhydride solution from the top; fresh solvent enters the vessel from below through a spray and removes from the water layer any acid or anhydride. C. HOLLINS.

Manufacture of aliphatic acid anhydrides. BRIT. CELANESE, LTD., H. DREYFUS, and W. BADER (B.P. 299,342, 23.7.27).—An acetate or acetic acid is heated with an alkali or alkaline-earth salt of meta- or pyrophosphoric acid at 150–180°, with or without the addition of inert diluents such as kieselguhr. E.g., a dry mixture of sodium metaphosphate, sodium acetate, and kieselguhr is passed through a reaction vessel at 150–180°, liquid glacial acetic acid being added at suitable intervals along its course; acetic anhydride (and acid) distils off, and the exhausted mixture, consisting of sodium orthophosphate etc., is regenerated by acidification and dehydration, and returned to the process.

C. HOLLINS.

Manufacture of crotonaldehyde and its homologues. DISTILLERIES DES DEUX SÈVRES (B.P. 274,488, 14.7.27. Belg., 14.7.26).—Aqueous acetaldehyde (50%) is aldolised at 15° or below in presence of alkali, the mixture is acidified (with phosphoric acid), and distilled, the crotonaldehyde being obtained in the form of its constant-boiling mixture with water, b.p. 84–85°. Butaldehyde under the same conditions gives Δ^{α} - α -ethylhexenaldehyde. C. HOLLINS.

Manufacture of dimethylolurea [bishydroxymethylcarbamide]. POLLOPAS, LTD., E. C. C. and E. J. BAILY (B.P. 299,487, 24.6. and 11.11.27).—Formaldehyde and carbamide are condensed below 35° in aqueous sodium hydroxide, the alkalinity being adjusted to about 0.01*N* so as to avoid rise of temperature. A bishydroxymethylcarbamide of m.p. 138–140° is readily obtained. C. HOLLINS.

Manufacture of compounds of the perylene series. I. G. FARBENIND. A.-G. (B.P. 272,528, 9.6.27. Ger., 9.6.26).—Perylenetetracarboxylic acid is decarboxylated in stages by heating it in neutral or alkaline aqueous suspension at 200–260°. The neutral potassium salt gives *perylene-3:9(or 10)-dicarboxylic acid*; in presence of potassium hydroxide a mixture of this and *perylene-3-carboxylic acid*, m.p. above 300°, results, whilst with larger excess of alkali perylene is obtained. The monoimide similarly yields with 7–8 equivalents of alkali *perylene-3:4-dicarboxylimide*, and with 2 equivalents *perylene-3-carboxylic acid*. C. HOLLINS.

Manufacture of highly-chlorinated perylenes. F. BENSA (B.P. 288,171, 1.3.28. Austr., 2.4.27).—In the presence of a chlorine carrier, such as aluminium chloride or antimony pentachloride, 7–10 atoms of chlorine may be introduced into perylene, e.g., by chlorination in nitrobenzene at 100–140°. C. HOLLINS.

Manufacture of sulphamic acids of aromatic secondary bases. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 298,550, 8.7.27).—Secondary arylamines (including diarylamines and carbazole) or mixtures containing them are treated with chlorosulphonic esters, sulphur trioxide, or pyrosulphuric acid in presence of an acid-binding agent (pyridine bases, sodium carbonate) with or without a solvent or diluent (pyridine bases, chlorobenzene) to give *N*-sulphonic acids, which may be purified and hydrolysed to regenerate the pure secondary base. C. HOLLINS.

Manufacture of phenylthioglycollic acids containing halogen and alkyl groups in the nucleus. I. G. FARBENIND. A.-G. (B.P. 281,290, 22.11.27. Ger., 24.11.26).—A halogenated toluene or xylene is treated with chlorosulphonic acid and the thiophenol obtained by reduction of the resulting sulphonyl chloride is condensed with chloroacetic acid. *m*-Chlorotoluene gives a 6-sulphonyl chloride, m.p. 54°, 5-chloro-*o*-thiocresol, and 5-chloro-*o*-tolylthioglycollic acid, m.p. 127°. 2:4-Dichlorotoluene-5-sulphonyl chloride, m.p. 71°, 4:6-dichloro-*m*-thiocresol, and 4:6-dichloro-*m*-tolylthioglycollic acid, m.p. 112°, are similarly obtained from 2:4-dichlorotoluene; 2:6-dichlorotoluene-3-sulphonyl chloride, m.p. 60°, the thiocresol, and 2:6-dichloro-*m*-tolylthioglycollic acid, m.p. 100°, from 2:6-dichlorotoluene; 2-chloro-*p*-xylene-5-sulphonyl chloride, m.p. 50°, 2-chloro-5-thio-*p*-xylenol, and 2-chloro-5-*p*-xylylthioglycollic acid, m.p. 96°, from 2-chloro-*p*-xylene. C. HOLLINS.

New intermediates and dyes therefrom. BRIT. DYESTUFFS CORP., LTD., and M. MENDOZA (B.P. 299,501, 27.7.27).—Amino- and diamino-diaryl sulphides are prepared by condensing 3-thiolsalicylic acids carrying a 5-substituent (e.g., methyl or chlorine) or a 5:6-benzo-group (as in 2-hydroxy-1-thiol-3-naphthoic acid)

with reactive halogenated aromatic nitro- or dinitro-compounds. Dyes showing little change in shade on chroming are obtained from the products (cf. B.P. 297,855; B., 1928, 887). 5-Chloro-3-thiolsalicylic acid, m.p. 198—200°, 5-thio-*p*-cresotic acid, m.p. 198°, and 2-hydroxy-1-thiol-3-naphthoic acid, decomp. 160°, are obtained by reduction of the corresponding disulphides, m.p. 250—252°, 249—250°, and 280° (decomp.), respectively, prepared from the sulphinic acids or sulphonyl chlorides. The dyes described are: 2-amino-2'-hydroxy-4-sulpho-3'-carboxy-5'-methylidiphenyl sulphide \rightarrow β -naphthol (scarlet), γ -acid (acid-coupled, blue-red), or 1-(2:5-dichloro-4-sulphophenyl)-3-methylpyrazolone (yellow); naphthionic acid \rightarrow 2:4-diamino-2'-hydroxy-3'-carboxy-5'-methylidiphenyl sulphide [red; the diamine, m.p. 178—180°, is prepared from the dinitro-compound, m.p. 27° (decomp.)]; benzidine = 2 mols. of the same diamine (deep brown); 2-amino-4-acetamido-2'-hydroxy-3'-carboxy-5'-methylidiphenyl sulphide \rightarrow γ -acid (red), R-acid (red-orange), or 1-(2:5-dichloro-4-sulphophenyl)-3-methylpyrazolone (yellow); 2-amino-2'-hydroxy-4-sulpho-3'-carboxy-5'-methylidiphenyl sulphide, or the 5'-chloro-compound, or 2-amino-4-sulphophenyl 2-hydroxy-3-carboxynaphthyl sulphide \rightarrow cresidine \rightarrow phenyl-J-acid (red-violet). C. HOLLINS.

Manufacture of alkylisorosindulinesulphonic acids. J. R. GEIGY A.-G. (B.P. 282,803, 28.12.27. Ger., 27.12.26).—By heating a 1-chlorodialkylisorosinduline acetate (from phenyl- β -naphthylamine and 3-chloro-4-nitrosodialkylaniline) with sodium hydrogen sulphite the chlorine is replaced by a sulphonic group and a second sulphonic group enters position 7, *para* to the nitrogen. The products are useful intermediates for naphthasafranine dyes. C. HOLLINS.

Manufacture of cyclic ketones. I. G. FARBENIND. A.-G. (B.P. 274,095, 7.7.27. Ger., 8.7.26. Addn. to B.P. 273,321; B., 1928, 887).—A hydrocarbon with free *peri*-positions is condensed with succinic anhydride or its mono- or *s*-dichloro- or *s*-dibromo-derivatives. Acenaphthene with succinic anhydride gives according to conditions 5- β -carboxypropionylacenaphthene, m.p. 206°, or the cyclic diketone, m.p. 180°. C. HOLLINS.

Manufacture of naphthalene-1:4:5:8-tetracarboxylic acid. I. G. FARBENIND. A.-G. (B.P. 274,103, 8.7.27. Ger., 9.7.26).—The cyclic diketone, m.p. 180°, of B.P. 274,095 (preceding) is oxidised with chromic-acetic or chromic-sulphuric acid. C. HOLLINS.

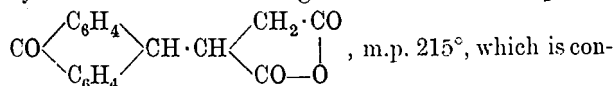
Intermediate compounds for sulphide dyes and dyes therefrom. IMPERIAL CHEM. INDUSTRIES, R. W. POPE, and M. WYLER (B.P. 299,152, 8.9.27).—Phenazines, naphthaphenazines, and dinaphthazines carrying a nuclear hydroxyl group are carboxylated with carbon dioxide or a bicarbonate; the products give on thionation sulphide dyes of increased redness and brightness. C. HOLLINS.

Production of dye intermediates [of the anthraquinone series]. W. SMITH, J. THOMAS, and SCOTTISH DYES, LTD. (B.P. 299,333, 19.4.27 and 9.1.28).—Chlorine or bromine in α -positions may be removed from halogenated β -aminoanthraquinones by reduction with alkaline glucose or hyposulphite or other agents capable

of producing a leuco-compound. *E.g.*, 1:3-dichloro-2-aminoanthraquinone is boiled with aqueous alkaline hyposulphite for 1 hr. and air-oxidised to give 3-chloro-2-aminoanthraquinone, m.p. 310°; 1-chloro-2-aminoanthraquinone yields 2-aminoanthraquinone.

C. HOLLINS.

Manufacture of benzantrones and intermediate products. I. G. FARBENIND. A.-G. (B.P. 277,342, 7.9.27. Ger., 10.9.26. Addn. to B.P. 268,830; B., 1928, 666).—Anthrone is condensed with maleic anhydride at 160—240° to give an additive compound,



verted by aluminium chloride in nitrobenzene at 100° into 3-hydroxybenzanthrone, m.p. 317°, with loss of carbon dioxide and hydrogen. By heating anthranol with maleic acid at 200° and then at 100—130° with sodium aluminium chloride, 3-hydroxydihydrobenzanthrone, readily oxidised on drying, is obtained. From anthrone and ethyl fumarate in sulphuric acid at 140° there is obtained a 3-hydroxybenzantronesulphonic acid. C. HOLLINS.

Manufacture of benzanthronecarboxylic acids. I. G. FARBENIND. A.-G. (B.P. 277,670, 14.9.27. Ger., 20.9.26).—3- or 4-Methylbenzanthrone is oxidised with nitrobenzene and potassium hydroxide at 140—150° to the corresponding 3-carboxylic acid, m.p. 335°, or 4-carboxylic acid, m.p. 307—308°. C. HOLLINS.

Manufacture of derivatives of pyrazolanthrone. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 298,775, 21.10.27).—Pyrazolanthrone is condensed with an aromatic halogen derivative in presence of copper powder, potassium acetate, and a solvent (nitrobenzene, amyl alcohol) to give *N*-derivatives. The following 2-substituted pyrazolanthrone derivatives are described: *phenyl*-, m.p. 211°; *o*-carboxyphenyl-, m.p. 262—265°; 3-carboxy-2-naphthyl-, m.p. 277—279° (decomp.); *o*-nitrophenyl-, m.p. 246—248°; 3-benzanthronyl-, m.p. 398—400°; 2-phenyl-3-benzanthronyl-; 9(10)-nitro-3-benzanthronyl-, m.p. 404—405° (amine, m.p. 417°); 4-benzanthronyl-, m.p. 398—400°; 1-anthraquinonyl-; 1-naphthyl-, m.p. above 300°. The 2-(3-benzanthronyl) derivatives of 5-methylpyrazolanthrone (m.p. 332—333°) and dipyrazolanthrone, and the product from pyrazolanthrone (2 mols.) and 1:3-dichloro-4:6-dinitrobenzene are also described. (Cf. B.P. 298,284; B., 1928, 923.) C. HOLLINS.

Production of complex metal alcoholates [ethoxides]. H. MEERWEIN, Assr. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,689,359, 30.10.26. Appl., 1.11.26. Ger., 12.11.25).—See B.P. 261,377; B., 1927, 316.

Production of alkylated aliphatic nitriles. M. BOCKMÜHL and G. EHRHART, Assrs. to WINTHROP CHEM. Co. (U.S.P. 1,690,325, 6.11.28. Appl., 17.6.26. Ger., 22.6.25).—See B.P. 253,950; B., 1927, 892.

Manufacture of dinitro-derivatives of perylene and its compounds. K. STIEGER, Assr. to F. BENSA (U.S.P. 1,688,837, 23.10.28. Appl., 5.1.26. Austr., 28.10.25).—See B.P. 260,568; B., 1927, 579.

Purification [of phthalic anhydride]. C. E. ANDREWS, Assr. to SELDEN Co. (U.S.P. 1,685,624, 25.9.28. Appl., 27.2.18).—See B.P. 142,902; B., 1920, 501 A.

Dinaphthylidicarboxylic acid and its manufacture. R. HERZ and W. ZERWECK, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,684,272, 11.9.28. Appl., 23.6.26. Ger., 2.7.25).—See B.P. 278,100; B., 1927, 902.

Furnace for catalytic gas reactions (F.P. 625,893).—See XI. **Phenols for germicides** (B.P. 299,522).—See XXIII.

IV.—DYESTUFFS.

Influence of sulphonie groups and other substituents on the colour of trisazo dyes. H. DINNEN (Z. angew. Chem., 1928, 41, 1078—1083; cf. Meuly, B., 1924, 86; Wanner, B., 1925, 537).—The absorption spectra of series of trisazo dyes of the Benzo Fast Blue type (sulphanilic acid \rightarrow α -naphthylamine \rightarrow Cleve acid \rightarrow J- or phenyl-J-acid) are examined by Formanek's method. In all examples the introduction of a new sulphonie group produces a bathochromic effect, which is usually smaller as more sulphonie groups are originally present. The effect is greater in *p*-position to the azo group than in *o*-position, and least in *m*-position, but is always small when the group is introduced into the first component. If α -naphthylamine is made the third component and Cleve acid the second, an appreciable bathochromic effect is observed; the position of the entering sulphonie groups often has greater effect than their number. Light-fastness is somewhat increased by the introduction of sulphonie groups; dyes having aniline-*o*-sulphonie or -2:4-disulphonie acid as first component are respectively faster than those derived from metanilic acid or aniline-2:5-disulphonie acid. If the first component is of the benzene series and the others are naphthalene derivatives, the dyes are faster to light than when the first two components are of the benzene series. The use of phenyl-J-acid in place of J-acid has a bathochromic effect, and light-fastness and affinity for cotton are increased; the solubility is diminished. The effect of the introduction of a methyl group may be batho- or hypso-chromic according to position, and is always small (positive in the first component, negative in the second); light-fastness is somewhat increased. Entry of an alkoxyl group, especially into the third component, produces a strong bathochromic effect; the difference between methoxyl and ethoxyl or propoxyl is inappreciable. The alkoxy-group improves the alkali- and acid-fastness of the disazo intermediate, but has little effect on that of the trisazo dye. C. HOLLINS.

Effect on coal-tar colours of lime and zinc white. ARNOLD.—See XIII.

PATENTS.

Manufacture of benzanthrone derivatives. BRIT. DYESTUFFS CORP., LTD., J. BADDILEY, A. SHEPHERDSON, and S. THORNLEY (B.P. 298,931, 16.7.27. Addn. to B.P. 276,766; B., 1927, 837).—The process of the prior patent is extended to simple homologues and derivatives, e.g., methyl- and chloro-benzanthrones.

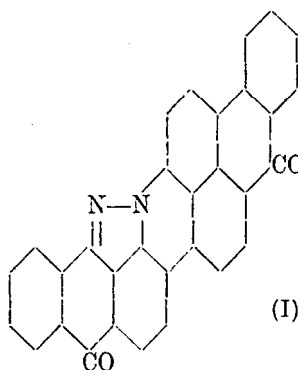
C. HOLLINS.

Manufacture of new vat dyes. W. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 298,583, 12.5.27).—The products obtained from 3-phenylbenzanthrone and aluminium chloride (B.P. 297,129; B., 1928, 847) are converted by alkaline fusion into bluish-green vat dyes. C. HOLLINS.

New black and grey vat dyes. BRIT. DYESTUFFS CORP., LTD., and S. THORNLEY (B.P. 298,928, 15.7.27. Addn. to B.P. 276,767; B., 1927, 837).—The process of the prior patent is extended to synthetic benzanthrone-sulphonic acids and to homologues and derivatives, e.g., methyl- and chloro-benzanthronesulphonic acids.

C. HOLLINS.

Manufacture of vat dyes. O. Y. IMRAY. From I. G. FARBERIND. A.-G. (B.P. 298,284, 12.7.27).—2-(3-Benzanthronyl)pyrazolanthrone (cf. B.P. 298,775; B., 1928, 922) is converted by alkaline fusion at 90—100°



(I)

into a blue vat dye (I), or at 160° into a greenish-grey to black vat dye (II). Other *N*-substituted pyrazolanthrone of B.P. 298,775 give greenish-blue to violet-blue vat dyes. Dichlorination of (I) with sulphuryl chloride or chlorine yields a violet-blue vat dye; a reddish-blue tetrachloro-compound and a greenish-blue dibromo-compound are also obtainable. Nitration of (I) for a bright green vat dye, becoming grey to black on chlorination, and of (II) for an olive-green (greyer on chlorination), is also described. The compound (I) is readily hydroxylated with sulphuric acid and manganese dioxide to a blue vat dye, which becomes greener and fast to chlorine on methylation. C. HOLLINS.

Manufacture of vat dyes of the anthraquinone series. J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 298,545, 7.6.27).—Anthraquinone-oxazoles containing as *C*-substituent an anthraquinonyl group are fast vat dyes of good affinity. Those carrying a free amino-group are deepened in shade by treatment with 65% oleum and a trace of sulphur, with or without subsequent methylation. Examples are: oxazole from 2:3-aminohydroxyanthraquinone and 2-aldehydoanthraquinone (yellow); from 2-amino-1-hydroxyanthraquinone and 1-amino-2-aldehydoanthraquinone (bluish-red); from 3-aminoalizarin and 1-amino-2-aldehydoanthraquinone, then methylated (red); from 1-amino-2-hydroxyanthraquinone and 1-aminoanthraquinone-2-carboxylic acid (bluish-red); from 1:3-dibromo-2-aminoanthraquinone and anthraquinone-2-carboxylic acid (yellow); from 2:3-bromoaminoanthraquinone and 1-aminoanthraquinone-2-carboxylic acid (bluish-red); from 2-amino-3-hydroxyanthraquinone and 1-amino-2-aldehydoanthraquinone, treated with oleum (blue) and methylated (violet); from 1:5-diamino-2:6-dihydroxyanthraquinone and 1-amino-2-aldehydoanthraquinone, treated with oleum (dark blue). C. HOLLINS.

Vat dye from dianthraquinonyldiaminophenanthraquinone. W. MIEG and A. JOB, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,684,327, 11.9.28. Appl., 28.6.26. Ger., 25.6.25).—2:7-Dibromophenanthraquinone is condensed with an α -aminoanthraquinone and the product is converted into a biscarbazole derivative by treatment with aluminium chloride or sulphuric acid. α -Aminoanthraquinone gives a red-brown vat dye, 1-amino-5-benzamidoanthraquinone a yellow-brown, 1-amino-4-benzamidoanthraquinone a red-brown (cf. B.P. 278,771 and 279,003; B., 1928, 9). C. HOLLINS.

Preparation of [sulphide] black colouring materials. R. VIDAL (B.P. 283,468, 9.1.28. Fr., 8.1.27).—2:4-Dinitro- or 2:4:6-trinitro-phenol or a mixture of these is first reduced with aqueous sodium sulphide, nitrosophenol or nitroso-*o*- or -*m*-cresol is added, and after reaction is complete the product is thionated with sodium polysulphide. The sulphide black is preferably precipitated by addition of an ammonium salt. C. HOLLINS.

Manufacture of sulphide dyes. A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 299,511, 28.7.27).—2-Nitroaceto-*p*-toluidide or 4-acetamido-*o*-toluidine is heated with benzidine (2 or more mols.) and sulphur at 200—260° for an orange sulphide dye. The 3-nitro-isomeride or its reduction product gives at 180—260° a greenish-yellow. C. HOLLINS.

Manufacture of [sulphide] dyes [from carbazole-indophenols]. SOC. CHEM. IND. IN BASLE (B.P. 280,595, 14.11.27. Switz., 13.11.26. Addn. to B.P. 199,360; B., 1924, 9. Cf. B.P. 270,348; B., 1927, 869).—The thionation of carbazole-indophenols for blue sulphide vat dyes is assisted by the addition of aliphatic compounds containing nitrogen and carbon in atomic ratio of at least 1:2, *e.g.*, carbamide, dicyanodiamide, semicarbazide, guanidine, thiocarbamide, ammonium thiocyanate, acetamide. Arylamines are not added. C. HOLLINS.

Polyarylamine dye and its manufacture. K. THIESS, B. DEICKE, and R. SCHMIDLIN, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,684,331, 11.9.28. Appl., 10.5.26. Ger., 22.5.25).—1:3-Dichloro-2:6-dinitrobenzene is condensed with an aminodiphenylamine-sulphonic acid (2 mols.) in boiling water in presence of alkali carbonate and a little alcohol to give wool dyes. 4'-Nitro-4-aminodiphenylamine-2'-sulphonic acid gives a yellow, 4-aminodiphenylamine-2-sulphonic acid a brown-yellow (claimed specially), 4-amino-4'-methyldiphenylamine-2-sulphonic acid an orange-yellow. 2:6-Dichloro-3:5-dinitrotoluene condenses with 4-aminodiphenylamine-2-sulphonic acid to give a brown. C. HOLLINS.

Water-soluble dye and its manufacture. O. SIEBERT, K. THIESS, B. SCHÖNER, R. SCHMIDLIN, W. BENADE, and B. DEICKE, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,684,330, 11.9.28. Appl., 17.6.26. Ger., 5.5.23).—See B.P. 274,999; B., 1927, 772.

Manufacture of sulphurised derivatives of phenols, and of naphthols. E. KRAUS, Assr. to FABR. VAN CHEM. PROD. (U.S.P. 1,690,640—1, 6.11.28. Appl., 16.2.26. Holl., [A] 5.8.25, [B] 24.11.25).—See B.P. 269,970; B., 1927, 469.

Manufacture of vat dyes derived from naphthalene-1:4:5:8-tetracarboxylic acid and aromatic *o*-nitroamines. W. ECKERT, Assr. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,690,775, 6.11.28. Appl., 27.1.27. Ger., 29.1.26).—See B.P. 265,232; B., 1928, 225.

Azo dye and its manufacture. E. HOFFA and E. FISCHER, Assrs. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,684,275, 11.9.28. Appl., 27.7.26. Ger., 1.8.25).—See B.P. 256,272; B., 1927, 837.

Manufacture of diphenyl- and ditolyl-methane [mordant] azo dyes. C. METTLER, Assr. to J. R. GEIGY A.-G. (U.S.P. 1,689,739 and 1,689,740, 30.10.28. Appl., [A] 14.1.27, [B] 25.11.27. Ger., [A, B] 28.1.26).—See B.P. 265,203; B., 1928, 442.

Dyes (B.P. 299,501). Sulphide dyes (B.P. 299,152).—See III. Lakes (B.P. 299,521).—See XIII.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Influence of temperature on sulphite-cellulose cooking. E. HÄGGLUND (Papier-Fabr., 1928, 26, 689—695).—If the conditions of cooking (circulation in the cooker etc.) are otherwise the same, defibreable cellulose in a yield of 55% of the weight of wood taken, with a 7—8% lignin content, may be obtained equally well at a maximum cooking temperature of 120° as at 135°. At the higher temperature decomposition takes place so rapidly that it is difficult to choose the right time for stopping the cooking, whilst at 120° no such difficulty is experienced, and in this lies the advantage of the lower temperature. For the stage of cooking corresponding with a bromine value of 0.15—0.30, *i.e.*, for a 52—48% yield of cellulose, it is immaterial whether the cooking is done at a maximum temperature of 120° or 135°, since the bromine values and yields of the products are identical; also, for the same degree of mealings, papers of equal strengths are given. For the "quick-cooking" process the yield of cellulose of all qualities is about 3% lower than for the longer processes at 120—135° maximum temperature, and an inferior product is obtained. Raising the cooking temperature 10° roughly doubles the velocity of decomposition, but variation of temperature has little effect on the acidity of the solution or on the sugar and sulphuric acid formation during cooking.

B. P. RIDGE.

Relation between the carbon, hydrogen, and oxygen contents in cotton cellulose under thermal decomposition, and its loss of weight. T. AKAHIRA (Sci. Papers Inst. Phys. Chem. Res., Tokyo, 1928, 9, 165—180).—Samples of absorbent cotton were heated for different times up to 13 days at constant or varying temperatures between 200° and 300° in air or nitrogen, under normal or reduced pressures, or in a high vacuum, and the carbon, hydrogen, and oxygen contents of the carbonised residues were found. Straight-line relationships which are independent of the heating conditions obtain between the percentage of each of the three elements and the percentage loss of weight of the original material. The ratio of hydrogen to oxygen in the residues is constant at 2:1 (*i.e.*, it is the same as for water or for the original cellulose molecule), and is

independent of the loss in weight of the material. From the curves obtained it is deduced that when the hydrogen or oxygen content is reduced to zero a certain value for the final carbon residue remains which approximates to 22.2%.
B. P. RIDGE.

Behaviour of lignin and chlorolignin in the preparation of wood pulp by means of chlorine. H. WENZL (Z. angew. Chem., 1928, 41, 1008).—In criticising the work of Waentig (cf. B., 1928, 476) the author describes processes using chlorine gas (Pomilio) and those using chlorine water (De Vains) as being fundamentally different, whereas Waentig regards these as chemically the same, but modified by the differences in temperature obtaining and the resulting effect on hydrochloric acid formation.

A. G. POLLARD.

Action of bromine on cellulose etc. FUCHS.—See II.

PATENTS.

Treatment of wood with steam for the manufacture of paper. R. BLASCHKE (F.P. 624,224, 11.7.27).—Wood is heated for 1–3 hrs. in steam under 5 atm. pressure, allowed to cool slowly for 5–10 hrs., and then treated with the usual chemicals for 3–10 hrs.

A. R. POWELL.

Weighting of silk. R. J. O'BRIEN, JUN., ASSR. to COLLWAY LABORATORIES, INC. (U.S.P. 1,684,286, 11.9.28. Appl., 16.12.24).—Silk threads are treated with rubber latex or solution and vulcanised. Tin oxide or zinc oxide may be added.

C. HOLLINS.

Cotton treating. A. COBB (U.S.P. 1,688,524, 23.10.28. Appl., 11.6.26).—Cotton is conditioned by being heated, while it is worked, to above 80° in a substantially dry atmosphere.

H. ROYAL-DAWSON.

Obtaining cellulose from ligno-cellulosic material. O. Y. IMRAY. From I. G. FARBENIND. A.-G. (B.P. 298,333, 30.8.27).—The comminuted raw material is treated with sufficient calcium hypochlorite solution containing not less than 2% of available chlorine (preferably about 2.8%) to oxidise the whole of the lignin. For fir wood that has been pretreated with, e.g., 0.5% caustic soda solution at 95°, about 50% of chlorine on the weight of wood is required. The temperature should not exceed 45° and the liquor is kept faintly alkaline during the process by the addition of an alkaline-earth hydroxide, preferably milk of lime; at no time, however, should the alkalinity be higher than is represented by the presence of 0.15% of free calcium hydroxide.

D. J. NORMAN.

Separating or extracting cellulose or paper pulp. (Sir) G. C. GODFREY, E. C. BENTHALL, E. S. TARTLTON, H. F. WHEELER [BIRD & Co.], and E. SPENCER (B.P. 291,064—5, 5. and 14.7.27. India, 28.5.27).—(A) In the semi-continuous, i.e., progressive, digestion of fibrous material such as bamboo, grasses, etc. the digesters are connected by a ring pipe provided with suitable cocks and valves so that the liquor in any one digester may be blown into any other digester in the ring. Preferably four or any larger even number of digesters are connected in a double ring, alternate digesters being connected to alternate rings so that the

digesters in one ring may be operated independently of those in the other ring. Only one ring pipe for steam and another for spent liquor need be provided for all the digesters in the system. (B) In order to avoid loss of cellulose during the final stage of progressive digestion processes, the concentration of active alkali should be highest at one of the intermediate lower-pressure stages, and the activity of digestion in the final stage should be modified by, e.g., reducing the concentration of the liquor, reducing the pressure, or replacing a part or the whole of the caustic soda in the liquor by sodium sulphide or sulphite.

D. J. NORMAN.

Manufacture of artificial [viscose] threads. L. LILIENFELD (B.P. 298,548, 6.5.27. Cf. B.P. 274,521; B., 1927, 745).—Viscose is spun into a solution, other than a strong mineral acid, which has both a coagulating effect and a swelling effect, or the viscose may be coagulated in an ordinary acid bath and then passed into a solution of any substance which has a swelling action on cellulose, e.g., zinc chloride in the presence or absence of mineral acids, cuprammonium hydroxide solution, strong organic bases, etc. The action of the swelling agent must be checked before it has any injurious effect on the thread. A 34% solution of zinc chloride in 20% sulphuric acid at 16° may be used as a coagulating and swelling bath, or the thread, after coagulation in an ordinary acid bath, may be passed into a solution containing 4200 pts. of zinc chloride, 500 pts. of concentrated hydrochloric acid, and 1800 pts. of water at 16°. The length of travel of the thread should be 10–20 cm. in the swelling bath and 120 cm. in air. The coagulated thread is wound on a bobbin the lower part of which is immersed in water.

D. J. NORMAN.

Paper pulp from cane (U.S.P. 1,688,904—5).—See XVII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Steeping and dyeing. R. LORENZ and F. MICHAEL (Z. physikal. Chem., 1928, 137, 142–175).—The tendency of wool and of proteins, such as animal membranes, to form compounds with hydrogen chloride has been determined at varying p_H values. For these substances the necessary conditions exist for the establishment of the Donnan membrane equilibrium, and by application of this theory the degree of swelling produced by the action of acids may be calculated. Indications may also be obtained of the process of adsorption of dyestuff anions, and its variation with the hydrogen-ion concentration of the bath, the presence of electrolytes, and the concentration of the dye. Experimental determinations of the optimum p_H for given dye concentrations are in good agreement with the theoretical values. The action of chromium mordants may be explained as due initially to hydrolysis of the chromium salt at the membrane; artificial loading of natural silk by steeping in stannic chloride solution depends on the same phenomenon.

H. F. GILLBE.

Effect of printing, discharging, and stripping agents on muslin. I. Rongalite groups and Decroline. II. Blankit, hyposulphite, sodium bisulphite, and steaming pressure. III. Ageing test. IV. Destruction of wool by hyposulphite groups.

H. UDAKA (J. Soc. Chem. Ind., Japan, 1928, 31, 374—381, 382—386, 386—388, 458—465).—I. The tensile strength and elongation of muslin decreased as the concentration of Rongalite C with zinc oxide and the time of steaming were increased. Zinc oxide promoted the lowering of the elongation of muslin and seemed to prevent the depression of the tensile strength. Distinct differences were observed between the effect of fresh Rongalite CW and that of a sample 1 year old. In the latter case the tensile strength and elongation were seriously affected. Decroline in acetic acid solution, as distinct from its aqueous solution, did not lower the tensile strength and elongation. Rongalite C and zinc powder had less effect than Rongalite C and zinc oxide.

II. Blankit and zinc oxide, and hyposulphite and zinc oxide both greatly decreased the tensile strength and elongation of muslin, but sodium bisulphite and zinc powder had little effect. These properties of muslin that had been treated with such reagents were also influenced by the steaming pressure used in printing.

III. Ageing slightly damaged muslin treated with the stable hyposulphite groups, but scarcely affected those treated with the unstable hyposulphite groups. The elongation of the treated muslin increased generally by ageing.

IV. The effects on wool of various decomposition products of hyposulphite groups and zinc salts were examined. Thus, zinc sulphate, sodium hyposulphite, and sodium bisulphate had little influence on wool fibre; sodium bisulphite was much more active. The Allwörden reaction was observed when wool fibres were treated with sulphuric acid; it is suggested that this reaction is caused by the hydrolysis of wool protein. Y. TOMODA.

PATENTS.

Dyeing etc. of materials made with or containing cellulose esters or ethers. BRIT. CELANESE, LTD., G. H. ELLIS, and H. C. OLPIN (B.P. 298,699, 19.7.27).—A suitable arylamine is oxidised on the fibre with *p*-toluenesulphonchloroamide or its sodium salt in presence of acid and/or an oxidation catalyst (e.g., vanadium chloride). C. HOLLINS.

Dyeing of materials made with or containing cellulose derivatives. H. C. OLPIN, ASST. to CELANESE CORP. OF AMERICA (U.S.P. 1,688,553, 23.10.28. Appl., 11.4.27. U.K., 9.2.27).—See B.P. 285,641; B., 1928, 364.

Dyeing, printing, or stencilling of acetyl cellulose or products made therefrom. G. H. ELLIS and W. O. GOLDTHORPE, ASSTS. to CELANESE CORP. OF AMERICA (U.S.P. 1,690,481, 6.11.28. Appl., 6.8.25. U.K., 14.8.24).—See B.P. 242,711; B., 1926, 50.

Dyeing leather with acid azo dyes. B. VOSSEN, ASST. to GRASSELLI DYESTUFF CORP. (U.S.P. 1,690,318, 6.11.28. Appl., 23.1.26. Ger., 3.2.25).—See B.P. 247,187; B., 1927, 329.

Apparatus for wet treatment of textile threads. M. SCHOENFELD (B.P. 298,721, 5.8.27).

[Supports for] fluid treatment of fabrics. BRIT. CELANESE, LTD. (B.P. 295,283, 7.8.28. U.S., 8.8.27).

Apparatus for dyeing of fabrics. C. HACKENBROICH (B.P. 295,569, 7.12.27. U.S., 13.8.27).

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Gas analysis in an Opl tower plant. W. HANSEN (Chem.-Ztg., 1928, 52, 813—815, 830—832).—For the analysis of exit gases a Riesenfeld meter is preferred to an aspirator. In this the gas is drawn through a capillary and the volume determined by the pressure drop. The maximum volume to be tested is 100—150 litres. The absorption train consists of a glass-wool filter, 2 bottles containing 50 c.c. of 0.1*N*-caustic soda, and 2 or 3 bottles containing 50 c.c. of acidified 0.5*N*-permanganate. No difference could be detected as a result of adding hydrogen peroxide to the caustic soda. Sulphuric acid alone is not a satisfactory absorbent for oxides of nitrogen, but mixed acid containing 5% of concentrated nitric acid gave good results, hydrogen peroxide in this case being added to the alkali absorbent. An examination of Raschig's method, using iodine and sodium acetate as absorbents, gave unsatisfactory results. Even with a gas velocity of 20 litres/hr., iodine was carried forward and the sodium acetate failed to retain nitric oxide. In addition, iodine is liberated from potassium iodide. The method is, however, suitable for tower gases containing sulphur dioxide in excess. It gave good results for the gases leaving tower 2, but not for gases later in the sequence. In such cases absorption in alkali and subsequent analysis of the absorbent is necessary. C. IRWIN.

Toxicity of fluorine compounds. S. MARCOVITCH (Tenn. Agric. Exp. Sta. Bull., 1928, No. 139, 1—48).—For insects and lower organisms, sodium fluosilicate is more toxic than sodium arsenite, possibly on account of their low calcium content; toxicity values are: sodium fluosilicate 34.5, sodium arsenite 13.1, sodium arsenate 4.8, sodium fluoride 4. For man and the higher animals the arsenicals are nine times as toxic as sodium fluosilicate, and 30 times as toxic as sodium fluoride. The protoplasmic poisoning action of fluorine is ascribed to the removal of calcium from the tissues as calcium fluoride. In presence of moisture, calcium hydroxide, when used as a carrier for sodium fluosilicate, may cause foliage injury. Calcium, magnesium, and strontium fluorides are only slightly toxic; of the organic acids, salicylic was the most toxic, followed by oxalic, formic, benzoic, and acetic acids. Of the metals, mercury was the most toxic; barium, lead, zinc, and copper were slightly toxic. The arsenicals and fluorine compounds are much more effective above 39°.

CHEMICAL ABSTRACTS.

Lead sulphate. H. SCHRADER (Farben-Ztg., 1928, 34, 268—269).—Lead sulphate is produced as a by-product of the manufacture of aluminium acetate for waterproofing tarpaulins etc. It may be used as a starting point for chrome yellow, first being converted into carbonate and then treated with potassium dichromate and sulphuric acid; the process has some advantage in the cheapness of lead as sulphate. Other variants of the method are: conversion of lead carbonate into basic nitrate or chloride by treatment with acid and double decomposition with alkali dichromate; decomposition of the lead sulphate with caustic soda or lime and treatment of the basic sulphate with dichrom-

ate; production of basic sulphate by treatment of the normal salt with dilute acetic acid and then proceeding as in the previous method.

C. IRWIN.

PATENTS.

Manufacture of sulphuric acid. W. A. PATRICK and E. B. MILLER, Assrs. to SILICA GEL CORP. (U.S.P. 1,683,694, 11.9.28. Appl., 24.5.24).—For production of an active catalyst a silica gel, capable when dry of absorbing at least 21% of its weight of water vapour at 30° and under 22 mm. partial pressure, is impregnated, while still containing 30–40% of water, with a solution of ammonium chloroplatinate and heated to 360°.

C. HOLLINS.

Manufacture of sulphuric acid. PULVERFABR. SKODA-WETZLER A.-G., and M. FEIGENSOHN (Austr. P. 106,847, 18.1.26).—The gases are passed, on their way between the Glover tower and the lead chamber, through a closed vessel in which they are brought into intimate contact with an oxidising medium, such as nitrous or nitric acid, which retains the selenium and arsenic present in the gases.

A. R. POWELL.

Preparation of purified phosphoric acid. J. MAXWELL, P. C. HOFFMANN, and C. E. MCCOY, Assrs. to VIRGINIA-CAROLINA CHEM. CORP. (U.S.P. 1,688,822, 23.10.28. Appl., 22.12.25).—Impure phosphoric acid is heated to 1200° or over in the presence of an oxidising gas, and the phosphoric acid is recovered from the fumes.

W. J. BOYD.

Manufacture of phosphoric acid and hydrogen. NORSK HYDRO-ELEKTRISK KVAELSTOF-A./S. (F.P. 624,438, 10.11.26. Nor., 23.12.25).—Phosphorus vapour and carbon dioxide are caused to interact at a high temperature, the phosphorus pentoxide formed is condensed and converted into phosphoric acid, and the carbon monoxide is mixed with steam and passed over a catalyst to obtain hydrogen and carbon dioxide for use again in the process.

A. R. POWELL.

Purifying brine to be used in electrolytic processes. J. DRUCKER, Assr. to I. G. FARBENIND. A.-G. (U.S.P. 1,687,433, 9.10.28. Appl., 17.8.27. Ger., 29.9.26).—The brine is saturated with an alkali fluoride, whereby any calcium or magnesium present is separated as flouride.

H. ROYAL-DAWSON.

Recovery of potassium chloride. N. M. McGRANE, Assr. to INTERNAT. PRECIPITATION Co. (U.S.P. 1,688,873, 23.10.28. Appl., 4.5.27).—Material containing potassium and sodium chlorides and sulphates is leached with an aqueous solvent at a raised temperature until the concentrations of the sodium and sulphate ions have reached, and then decreased considerably below, their maximum values, while the concentrations of the potassium and chlorine ions have continued to increase. The solution is then separated from undissolved solid and cooled to cause deposition principally of potassium chloride crystals.

L. A. COLES.

Manufacture of potassium nitrate. WOLFF & Co., and F. FROWEIN (Austr. P. 106,983, 25.5.26. Ger., 25.5.25).—Nitric acid is neutralised with powdered dolomite and the clarified solution is treated with potassium sulphate. The heat of the reactions is

utilised to obtain hot, highly concentrated solutions from which potassium nitrate crystallises on cooling.

A. R. POWELL.

Production of nitrites. E. T. DRAKE, Assr. to CUDAHY PACKING Co. (U.S.P. 1,685,629, 25.9.28. Appl., 8.2.26).—An aqueous solution of sodium nitrate, sugar, common salt, and amino-nitrogen is inoculated with selected groups of nitrate-reducing and salt-tolerant bacilli.

C. RANKEN.

Recovery of alkali chlorides from minerals. A. J. MACDOUGALL (Can. P. 265,449, 10.11.25).—Lithium and potassium chlorides are recovered from aluminosilicates, such as lepidolite, spodumene, petalite, etc., by heating the powdered mineral mixed with carbon in a current of chlorine.

A. R. POWELL.

Preparation of pure alumina. ZAIDAN HOJIN RIKAGAKU KENKYUJO (B.P. 284,661, 29.7.27. Jap., 3.2.27).—Clay or bauxite is reduced with carbon in an electric furnace and silicon, iron, ferrosilicon, and iron oxides are removed from the resulting impure alumina by treatment with chlorine or hydrogen chloride at 200° or above, with the addition of a reducing agent if iron oxides are present.

W. G. CAREY.

Production of compounds of metal and sulphur. K. BRODOWSKI (B.P. 290,986, 21.5.28. Ger., 21.5.27).—The appropriate metal and sulphur are heated under pressure by the passage of an electric current with exclusion of oxygen, and the heated mass is forced into metal moulds lined with insulating material, e.g., paper.

W. G. CAREY.

Manufacture of hydrogen. G. A. MOURLAQUE (F.P. 624,330, 22.9.26).—Steam is generated by means of an electrical resistance heater arranged inside a porous cylinder, and is then caused to react with a suitable metal for the production of hydrogen.

A. R. POWELL.

Purification of gases. J. Y. JOHNSON, From I. G. FARBENIND. A.-G. (B.P. 298,726, 10.8.27).—Impurities injurious to catalytic reactions are removed from gases by treatment, preferably at an elevated pressure, with reactive metals dissolved or finely distributed in melts of salts, oxides, or hydroxides, or melts of other metals, to which may be added substances inert towards the melts but having a catalytic effect on the purifying reaction. Any metallic vapour may be recovered from the gas by means of active charcoal.

H. S. GARLICK.

Lead carbonate from ores (N.P. 41,124).—See X. **Barium compounds for sugar manufacture** (U.S.P. 1,688,071).—See XVII.

VIII.—GLASS; CERAMICS.

Boric oxide-silica glasses. A. COUSEN and W. E. S. TURNER (J. Soc. Glass Tech., 1928, 12, 169–190).—Clear boric oxide-silica glasses were prepared up to a silica content of 80%, and homogeneous samples were obtained over the range 0–61.4% SiO₂. The thermal expansion of boric oxide and of the mixtures was measured up to the softening point. Continuous diminution of the rate of expansion (which was linear up to the lower critical point) occurred with increase of silica content.

In addition, the lower and upper critical temperatures continuously increased, and the straight portion of the curve in the critical range became progressively shorter. The density of pure, annealed boric oxide was found, as a mean of two methods, to be 1.8442, the values previously given by Briscoe, Robinson, and Stephenson (B., 1926, 219) being too low, principally through lack of annealing. Densities of the silica-boric oxide glasses continuously increased with increase of silica content, the values lying on a smooth curve. A. COUSEN.

Determination of the durability of glass by the powder method. M. THOMAS (J. Soc. Glass Tech., 1928, 12, 87—92 r).—A standard method, chosen by a Committee of the German Society of Glass Technology, is a compromise, due to practical considerations, between the more desirable measurement of deep-seated as opposed to surface durability. By it, 10 g. of glass of grain size 0.30—0.49 mm. are boiled for 5 hrs. with 100 c.c. of water, in a salt-water bath at 100°. A. COUSEN.

Thermal expansion factor of aluminium oxide in glass. I. KITAIGORODSKY and S. RODIN (J. Soc. Glass Tech., 1928, 12, 164—168).—Dilatometric determinations of the thermal expansions of glasses rich in alumina gave results deviating but little from theoretical values calculated from the factors given by English and Turner (B., 1928, 262), and confirmed, in particular, the factor for alumina, *i.e.*, 0.52, as opposed to the value 5.0 previously given by Winkelmann and Schott. A. COUSEN.

Characteristics of refractory clays for the glass industry. G. GEHLHOFF, H. KALSING, K. LITZOW, and M. THOMAS (J. Soc. Glass Tech., 1928, 12, 213—279).—Results are given of the analyses and determination of properties of a number of German and Bohemian clays, some kaolins, natural sillimanite, pure alumina, and Dinas brick. Properties studied were: (1) the proportion of water needed to produce working plasticity, (2) drying and firing shrinkage, (3) bending strength, (4) porosity, (5) fusion temperature, (6) softening under load, (7) thermal resistance, (8) resistance to corrosion by batch materials, molten batch, and glass, both quiescent and in motion. Chemical resistance and stability of the clays at high temperatures increased with increase of alumina content (except to attack by saltcake). They depended also on the firing temperature, and in particular on density of burning and porosity. Grog grains under 2 mm. in diam. did not influence the softening temperature under load, but affected the resistance to chemical attack. Compounds in clays, such as iron oxide and alkalis, in certain cases injuriously affected the properties, irrespective of the alumina content. A. COUSEN.

Expansion and tensile tests on glass-house refractories. J. F. HYSLOP, R. F. PROCTOR, and H. C. BIGGS (J. Soc. Glass Tech., 1928, 12, 190—204).—From expansion tests up to 1400°, silica-alumina refractories were classified into three groups according as they showed (1) rapid and large contractions beginning at, or above, 1000° (characteristic of lightly fired ware), (2) a decreased rate occurring gradually or abruptly above 1100° (hard-burnt clays), and (3) a more or less uniform rate of expansion up to 1400° (hard-burnt

sillimanite and synthetic silica-alumina materials. Tensile tests on pot clays show the good effect of hard burning and the close relationship between the condition of the grog and the resistance to stress at high temperatures. For good and complete burning, aluminous clays are superior to siliceous, whilst the presence of iron oxide lowers the temperature of rupture.

A. COUSEN.

PATENTS.

Tunnel kiln, furnace, etc. C. F. GEIGER, Assr. to CARBORUNDUM Co. (U.S.P. 1,686,083, 2.10.28. Appl., 1.2.27).—The kiln is provided with a combustion chamber which is movable along the whole length of the tunnel. A. R. POWELL.

Drying ceramic ware and apparatus therefor. T. H. RHODES, Assr. to PROCTOR & SCHWARTZ, INC. (U.S.P. 1,689,082, 23.10.28. Appl., 13.3.24).—The articles are passed through a long drying chamber into which currents of air increasing in temperature towards the discharge end are introduced at points along the chamber, and are passed transversely across it and caused to blend with one another so as to maintain a uniform increase in temperature along the chamber. L. A. COLES.

[Porcelain] insulators. S. E. AUSCHER (F.P. 620,987, 30.9.25).—The slip used for making ordinary porcelain, which is fired at 1400°, is mixed with a proportion of finely-ground porcelain which has already been fired at its usual firing temperature. The mixture is cast into shapes, fired, and glazed as usual; the products have great strength and a high dielectric constant. A. R. POWELL.

Manufacture of rubber-bonded abrasive articles. D. E. WEBSTER, Assr. to NORTON Co. (U.S.P. 1,687,410, 9.10.28. Appl., 30.10.25).—A mixture of rubber latex, a vulcanising agent, an accelerator, and abrasive grains is coagulated and vulcanised. H. ROYAL-DAWSON.

Insulators (B.P. 299,408).—See XI.

IX.—BUILDING MATERIALS.

Effects of moisture changes on building materials. R. E. STRADLING (Dept. Sci. Ind. Res. Building Res. Bull. No. 3, 1928, 22 pp.).—The effects of continuous or intermittent wetting of building materials resulting eventually in their disintegration may be due to actual chemical combination in the material, *e.g.*, the hydration of a grain of lime near the surface, or by dissolution of constituents, *e.g.*, of lime by sulphur acids in rain with the production and destructive crystallisation of calcium sulphate. The alternation in the formation of ice crystals during winter is not considered vitally destructive in England, though serious damage has been observed in Sweden from this cause, and it is doubtful whether osmosis can exert sufficient pressure owing to the nature of the materials to cause disruption of a building. It has been shown that repointing old buildings with a strong mortar has sometimes been damaging in effect due to soluble salts being forced afterwards through the brick or stone work, whereas formerly they escaped through the softer mortar joint. The effect of "sorbed" water is unknown, though many

materials possess the property of absorbing water, causing the swelling of a gel formation. With increasing moisture content the stress-strain values of Yorkshire sandstones decreased, though corresponding values for cement concrete were less regular, due probably to the effect of internal strain in the cementing material when drying-out takes place. Some correlation has been obtained between the measurement of the adsorption of water and the adsorption of dyes. C. A. KING.

Thermal constants and strength of concrete of light aggregates. M. HAMADA (J. Fac. Eng. Tokyo Imp. Univ., 1928, 17, 201—212).—Tests on cylinders made from Japanese materials and of the size used for the standard compression test showed that for each kind of aggregates the mixture and age have comparatively little effect on the diffusivity of concrete, though the nature of the aggregate caused a wide difference, concrete composed of common sand and gravel showing the largest value. Soft "Kokaseki," a kind of liparitic pumice, and lapilli gave a diffusivity value of only about 50% of that of ordinary concrete; hard Kokaseki and cinders about 60%; and broken brick 65%. The apparent sp. gr. increased with the relative richness of cement in the mixture; soft Kokaseki had the lowest value of all aggregates. Little variation (0.2—0.3) was observed in the sp. heats of the specimens, but the lowest value for conductivity (0.0012) was obtained with Kokaseki concrete. All the light concretes showed very low strength, the decrease in strength being greater than the decrease in weight. C. A. KING.

Thermal conductivities of walls, concretes, and plasters. E. GRIFFITHS (Dept. Sci. Ind. Res. Building Res. Tech. Paper No. 6, 1928, 19 pp.).—A presentation of the methods used for determining the heat-transmission of materials commonly used for building purposes. C. A. KING.

PATENTS.

Production of porous concrete etc. AEROCRETE (FOREIGN), LTD., and C. NICOL (B.P. 299,484, 28.4.27).—Material for the production of porous concrete comprises Portland cement etc., zinc or aluminium powder, and liquid or solid grease, *e.g.*, stearin, which is preferably mixed previously with the metal powder in the absence of air. L. A. COLES.

[Cement for] grinding stones or wheels. BIRMINGHAM SMALL ARMS CO., LTD., A. R. PAGE, and P. S. DEVEREUX (B.P. 299,633, 16.12.27).—A cement which sets without heating for filling spaces between the segments of abrasive material comprising grinding stones is made from abrasive grit 100 pts. by wt., calcined magnesium oxide 15 pts., and a saturated solution of magnesium chloride 35 pts. W. G. CAREY.

Material for damping sound and other waves. M. HAHN and K. B. EISENBERG (B.P. 299,543, 9.8.27).—Acoustic, mechanical, electrical, and thermal waves are damped by applying to walls etc. a coating comprising a non-drying oil, *e.g.*, mineral oil, and powdered filling material, *e.g.*, loam or clay. L. A. COLES.

Production of calcium aluminate cement. E. M. ROCHE, Assr. to U. B. VOISIN (U.S.P. 1,689,891, 30.10.28.

Appl., 4.12.24. Fr., 6.5.24).—See B.P. 233,698; B., 1925, 960.

Bituminous supports (B.P. 299,208).—See II.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Relation between the quantity and depth of carburisation [of iron]. G. TAKAHASHI (Sci. Rep. Tōhoku Imp. Univ., 1928, 17, 1135—1156).—The relation between the amount of carbon which diffuses into iron and the depth of the diffusion zone has been determined for various carburising reagents at temperatures between 880° and 1000°. In all cases the weight increment-depth of penetration graphs are parabolas for any given temperature. The more efficient the carburising agent the greater is the amount of carbon absorbed for a given depth of carburisation. Wood charcoal is a more efficient carburising agent than any other form of solid carbon, but its efficiency is greatly reduced by prolonged heating at 900—1000° prior to use. On the other hand its efficiency is greatly increased by admixture with alkali or alkaline-earth carbonates. With these mixtures the quantity of carbon absorbed for a given depth of penetration is the greater the higher is the temperature; with carbon monoxide or a mixture of hydrogen and ethylene or acetylene, however, the quantity of carbon absorbed at 900—1000° is the greater the lower is the temperature or the longer the time. When the carburising temperature is between 900° and the A1 point, the percentage of carbon in the innermost layer is the greater the lower is the carburising temperature. A. R. POWELL.

Corrosion of iron. H. ENDŌ (Sci. Rep. Tōhoku Imp. Univ., 1928, 17, 1111—1134).—The loss in weight of Arceo iron in distilled water is a linear function of the time, and is greater in sunlight than in the dark; the rate increases with rise of temperature to 60—70°, then decreases owing to the smaller solubility of oxygen in water at high temperatures. In nitric acid solutions the rate of dissolution increases with rise of concentration up to 30%, then falls almost to zero in 50% nitric acid; in hydrochloric acid solution the rate of dissolution increases very rapidly when the concentration exceeds about 15%, whereas in sulphuric acid solution the rate of dissolution is almost a linear function of the concentration. The rate of corrosion of iron in various salt solutions has been determined. Oxidising salts inhibit the rusting of iron when their concentration exceeds a certain limiting value; for potassium chromate and dichromate this concentration is 0.0001*M*, for potassium chlorate 0.01—0.003*M*, and for potassium permanganate 0.002—0.001*M*. For most other salt solutions corrosion increases rapidly to a maximum with increasing concentration, then falls much more slowly. Addition of potassium chromate to chloride solutions greatly retards their corrosive action on iron, whereas the dichromate tends to increase the action owing to the liberation of hydrochloric acid. A. R. POWELL.

Protection of steel against oxidation at the end of the open-hearth process, and decrease in the amount of gas occluded. V. N. TSVIBEL (Tekh.

Proizvodstvo, 1927, 36—49).—It is proposed to protect the slag from the action of the oxidising atmosphere by the use of a layer of heavy hydrocarbons.

CHEMICAL ABSTRACTS.

Properties of quenched steels. K. TAMARU (Bull. Inst. Phys. Chem. Res., Japan, 1928, 7, 1028—1034).—The magnitude of the A0 transformation was found to be large when the separated cementite was fine, and to become smaller as it grows. Pearlite cementite showed the largest transformation, and globular cementite the least. Two magnetic maxima were observed, at 120° and at 300—350°, when quenched high-carbon steel was tempered at gradually increasing temperatures, and both were considered to be due to the decomposition of β -martensite and the growth of minute cementite particles. Quenched steel contains α - and β -martensites. The former is more expanded than the latter, and is transformed into it at 100—150°; a quenched steel therefore contracts up to 200° and has a density greater than the original but smaller than annealed steels. Above this temperature the quenched steel expands to 260°, due to the decomposition of austenite into α -martensite and then into β -martensite, and above this temperature it contracts, due to the decomposition of β -martensite into troostite. F. S. HAWKINS.

Rapid determination of alloying constituents in iron by spectrographic analysis. G. SCHEIBE and A. NEUHÄUSSER (Z. angew. Chem., 1928, 41, 1218—1222).—The method of determining the percentage of manganese and silicon in iron by measuring the relative intensities of two neighbouring spectral lines, one due to iron and one to the alloying element, is described, and curves are given showing the variations in the intensity ratios Si/Fe and Mn/Fe for several pairs of lines when alloys of varying silicon and manganese content are examined. The capacity and self-inductance of the spark circuit must be standardised by comparing the intensities of the two tin lines, 3331 Å. and 3352 Å., and standardised photographic plates must be used throughout the tests. Once a series of curves is obtained for several pairs of lines by spectrographing standard alloys, the determination of silicon or manganese in iron may be made in 15—20 min. with an accuracy of $\pm 0.1\%$.

A. R. POWELL.

Influence of cobalt on determination of manganese in steel. I. WADA and S. SAITO (Bull. Inst. Phys. Chem. Res., Japan, 1928, 7, 1002—1027).—Cobaltous nitrate is oxidised by sodium bismuthate in the presence of nitric acid, and in the presence of ferric nitrate permanganates are reduced. Hence the manganese in a sample of steel cannot be determined by dissolution in nitric acid, oxidation to permanganate by sodium bismuthate, and titration with ferrous ammonium sulphate if cobalt be present. In the absence of iron, manganese can be determined by this method with cobalt present.

F. S. HAWKINS.

The hydrogen-antimony-tin method for the determination of oxygen in cast iron. B. KJERMAN and L. JORDAN (U.S. Bur. Stand. Res. Paper No. 25, 1928, 20 pp.).—The method of Oberhoffer and others (cf. B., 1925, 993) has been examined, and it is shown that for precisions of the order of 0.006% of oxygen the results compare favourably with those given by the

vacuum-fusion method (cf. Jordan and Eckman, B., 1926, 94), but, on account of its greater precision, the latter method is preferred for cast iron containing 0.01—0.04% O. The method is applicable to alloys of high silicon content provided that the content of carbon is also high. In both methods lump rather than milled samples should be used, as adsorption of air and moisture and surface oxidation lead to high results with the latter. M. E. NOTTAGE.

Rapid analysis of bronze and brass without electrolysis. H. KRUG (Chem.-Ztg., 1928, 52, 842).—The alloy (5 g.) is dissolved in 40 c.c. of nitric acid (d 1.4), 200 c.c. of boiling water and 3 g. of ammonium nitrate are added, and the solution is filtered into a 500 c.c. graduated flask, the precipitate of metastannic acid being washed, ignited to stannic oxide, and weighed. After dilution to the mark, 100 c.c. of the filtrate are treated with sulphuric acid for the determination of lead as sulphate, a second portion of 100 c.c. is neutralised with ammonia and the manganese precipitated as dioxide by boiling with ammonium persulphate, and a third portion is treated with ammonia, the washed precipitate dissolved in sulphuric acid, the solution passed through a Jones reductor filled with cadmium filings, and the ferrous sulphate formed titrated with permanganate. Copper may be determined in the filtrate from the lead sulphate or, if the lead and iron content of the metal is low, 25 c.c. of the original solution are neutralised with ammonia, the copper and iron precipitated by addition of disodium hydrogen phosphate, and the copper is redissolved in 10 c.c. of 50% acetic acid and determined iodometrically. Zinc is determined as pyrophosphate in a further 50 c.c. of the original solution after removal of copper and lead by hydrogen sulphide and of manganese and iron by ammonium persulphate and ammonia. A. R. POWELL.

Thermal expansion of magnesium and some of its alloys. P. HEDNERT and W. T. SWEENEY (U.S. Bur. Stand. Res. Paper No. 29, 1928, 22 pp.).—Data for the linear thermal expansion of cast and extruded magnesium between -183° and 500° , for cast and extruded magnesium-aluminium alloys containing up to 10.4% Al, and for magnesium-aluminium-manganese alloys containing up to 4.1% Al and 0.9% Mn, have been obtained, three types of apparatus being used. The following average equation represents the expansion of magnesium between 20° and 500° : $L_t = L_0 [1 + (24.80t + 0.00961t^2) \times 10^{-6}]$. The coefficients of expansion for the extruded materials are slightly less than for cast materials. Practically no change in the coefficient is caused by the addition of up to 10% of aluminium to magnesium, but additions of 0.9% of manganese causes a slight increase and of 0.3% a slight decrease. M. E. NOTTAGE.

Function of oxygen in the dissolution of metals and minerals by cyanide solutions. R. HAY (J. Roy. Tech. Coll. Met. Club, Glasgow, 1927—8, [6], 23—28).—Experiments on the rate of dissolution of gold, silver, copper, and lead in dilute cyanide solutions indicate that the primary action is associated with the formation of a film of hydrogen on the surface of the metal, and this film retards further action unless it is removed by means of a depolariser such as atmospheric oxygen or by contact

with another metal. Thus copper does not dissolve in unacrated cyanide solutions except when in contact with lead; dissolution then proceeds rapidly with evolution of hydrogen. In the precipitation of gold from complex cyanide solutions nascent hydrogen is the active agent, and this is formed by the action of the zinc on the free cyanide in the solution. This action is accelerated by the use of a zinc-lead or zinc-copper couple or by using zinc with ragged edges so as to reduce the overpotential of the hydrogen.

A. R. POWELL.

Electrodeposition of chromium. J. G. ROBERTS (J. Roy. Tech. Coll. Met. Club, Glasgow, 1927—8, [6], 19—22).—A review of recent work.

A. R. POWELL.

Mineral separation in a finely-divided state. R. C. EMMONS (Amer. J. Sci., 1928, 16, 423—445).—Experiments are described on the separation of minerals by means of the radiometer effect. The finely-powdered mineral was allowed to fall through a beam of light in a closed vessel containing gas at a low pressure.

C. W. GIBBY.

PATENTS.

Operating shaft furnaces, particularly blast furnaces. F. KRUPP A.-G. FRIEDRICH-ALFRED-HÜTTE (B.P. 275,601, 25.7.27. Ger., 6.8.26).—When using a portion of waste furnace gases in operating a blast furnace, the blast is blown at a higher temperature to compensate for the loss of heat at the hearth or other particular zone of the furnace, whilst the coke charge is correspondingly reduced. The thermal and chemical equilibria may be compensated also by supplying oxygen or by the introduction of coal dust close to the tuyères.

C. A. KING.

Operation of open-hearth furnaces. F. B. MCKUNE, Assr. to OPEN-HEARTH COMBUSTION CO. (U.S.P. 1,687,682, 16.10.28. Appl., 5.5.22).—A mixture of air and fuel is fed into a furnace chamber through a relatively constricted inlet, and additional air is supplied through another inlet; the products of combustion are delivered through these inlets and additional areas, these together forming an outlet of greater area than the combined area of the inlets.

J. S. G. THOMAS.

[Annealing] furnace. A. N. OTTS and L. F. WOOLSTON, Assrs. to GEN. ELECTRIC CO. (U.S.P. 1,688,745, 23.10.28. Appl., 12.3.27).—A preheating chamber attached to the main furnace contains a vertically movable support to receive the cold charge for preheating and the heated charge for cooling.

L. A. COLES.

Bright-annealing furnaces. SIEMENS-SCHUCKERTWERKE A.-G., Assees. of SIEMENS-SCHUCKERTWERKE GES. M.B.H. (B.P. 282,079, 8.12.27. Ger., 9.12.26).—The protective gas supplied to a bright-annealing furnace is preheated by being passed between the arched cover of the furnace and an internal "false" cover, and then through openings in the furnace walls. The gas supply pipe may be heated electrically.

C. A. KING.

Electric [arc] furnace [for steel]. A. E. GREENE (U.S.P. 1,687,936, 16.10.28. Appl., 18.5.25).—Silica sand is added to a bath of molten steel covered with slag under an electrode on top of the slag, and a carbonaceous reducing agent is added on top of the sand. The

mixture is heated by the arc until the silica is reduced and the steel is deoxidised and finished.

J. S. G. THOMAS.

Refining iron and steel. C. W. HILL (U.S.P. 1,686,087, 2.10.28. Appl., 30.11.25).—The metal is refined by means of a current of suitable gas passed into the molten metal under such conditions of temperature and pressure that the amount adsorbed by the metal may be controlled at will.

A. R. POWELL.

Making malleable-iron castings. H. A. SCHWARTZ, Assr. to NAT. MALLEABLE & STEEL CASTINGS CO. (U.S.P. 1,688,438, 23.10.28. Appl., 23.12.26).—A casting of usual composition made in a sand mould is cooled rapidly from above the critical point, then heated above the critical point until cementite is broken down, and cooled.

C. A. KING.

Hard solder for grey pig iron, cast steel, iron, etc. METALLBANK U. METALLURGISCHE. GES. A.-G. (B.P. 285,485, 13.2.28. Ger., 18.2.27).—The solder consists of 42—60% Cu, 1—10% Ni, and up to 3% Si, the remainder being zinc.

H. ROYAL-DAWSON.

Carburisation of ferrous metals. BRIT. THOMSON-HOUSTON CO., LTD., Assees. of G. W. HEGEL and G. R. BROPHY (B.P. 276,675, 25.8.27. U.S., 26.8.26).—The metal is heated in an atmosphere produced by the vaporisation of a mixture of 4 pts. of bone oil, 4 pts. of kerosene, and 1 pt. of nitrobenzene, the last-named compound acting as an energetic accelerator of carburisation.

A. R. POWELL.

Rust-proofing [of iron and steel]. PARKER RUST PROOF CO., Assees. of M. GREEN and H. H. WILLARD (B.P. 270,679, 25.4.27. U.S., 10.5.26).—Iron or steel articles are rendered non-rusting by immersion in a hot solution of phosphoric acid containing ferrous and manganous dihydrogen phosphate in such proportions that the iron content is 2—3 times the manganese content of the solution.

A. R. POWELL.

Apparatus for reducing oxides of metals [copper, lead, or zinc]. J. W. HORNSEY (B.P. 277,325, 5.9.27. U.S., 8.9.26).—The apparatus comprises a vertical shaft connected at its upper end with a stack and at its lower end with a dust-collecting device. A rotating, preheating cylinder opens into the lower half of the shaft and is slightly inclined downwards away from the shaft towards a second rotating cylinder, which acts as the reducing chamber, and which is connected to the preheating chamber through a stationary structure in which the two cylinders rotate on gas-sealed bearings. The hot gases from the reducing chamber are ignited in their passage into the preheating chamber so as to preheat the materials (finely-divided coal and ore) therein. A water-cooled screw conveyor serves to assist the passage of the charge through the apparatus and to keep it thoroughly mixed.

A. R. POWELL.

Reduction of zinciferous materials. L. MELLERSH-JACKSON. From NEW JERSEY ZINC CO. (B.P. 298,921—4, 8.7.27).—(A) The reduction is carried out in a continuously operating vertical retort into which is charged a mixture of sintered lumps of zinciferous material and briquettes made by coking a mixture of bituminous coal and roasted zinc ore. The charge is made of such porosity

that the heat applied to the outer walls of the retort is conducted throughout the mass by the convection of currents of gas heated at the walls. (b) The retort is made of a non-scaling metal such as nickel-chromium, nickel-iron, chromium-iron, or iron-nickel-chromium alloy, and the temperature of the outer walls is maintained at about 1150°. Provision is made for the expansion and contraction of the retort and for maintaining a reducing atmosphere therein. (c) The retort is lined with refractory bricks with a carborundum base fitted together with butt joints cemented with a thin layer of refractory material. (d) The condenser is connected at its upper end with a stack for the removal of waste gases, the position and height of the stack being so arranged as to give sufficient draught through the retort to prevent the gaseous products of the reaction from escaping through the discharge opening at the lower end of the retort while the residues from the distillation are being removed from the retort. A. R. POWELL.

Reduction of zinc ores. FASTIGHETS AKTIEBOLAGET ØRESUND (N.P. 40,950, 11.3.21).—The mixture of ore and reducing agent is preheated in an electrically heated rotating furnace prior to treatment in the reducing furnace. A. R. POWELL.

Continuous manufacture of lead carbonate or of mixtures of lead carbonate and zinc oxide directly from ores. TROLLHÄTTANS ELEKTROTHERMISKA AKTIEBOLAG (N.P. 41,124, 28.7.22).—The finely-divided ore is reduced in an electric furnace at a sufficiently high temperature to cause volatilisation of the lead and zinc, and the metal vapours are treated with a mixture of oxygen and carbon dioxide. A. R. POWELL.

Treatment of cupriferous molybdenite. NORSKE MOLYBDAENPRODUKTER A./S. (N.P. 41,615, 31.3.24).—The ore is subjected to a flotation treatment and the concentrates are run over a rapidly vibrating shaking screen under a spray of high-pressure water, whereby the copper pyrite is washed through the screen, leaving the molybdenite flakes on the surface. A. R. POWELL.

Purification of metals [copper]. H. H. ALEXANDER (U.S.P. 1,687,277, 9.10.28. Appl., 28.10.25).—The copper is melted by contact with combustion gases the composition of which is continuously regulated by controlling the air and fuel supplies, in accordance with the impurities present in the copper. F. G. CLARKE.

[Silicon]-copper alloys. METALLBANK U. METALLURGISCHES GES. A.-G. (B.P. 288,974, 5.4.28. Ger., 16.4.27).—Silicon-copper alloys containing 0.5–10% of one or more of the metals nickel, cobalt, manganese, iron, or chromium, together with 0.5–10% of lead, cadmium, or thallium, or mixtures of these are claimed. A. R. POWELL.

Copper-silicon-manganese alloys. AMER. BRASS CO. (G.P. 450,278, 11.10.24).—Acid-resisting copper alloys contain 92.3–96.5% Cu, 3–6.5% Si, and 0.5–1.2% Mn. A. R. POWELL.

Manufacture of white alloys. A. L. POCOCK (B.P. 299,493, 25.7.27).—An alloy of zinc with 5–15%

of an alloy of 2 pts. of copper, 4 pts. of aluminium, and 1 pt. of silver is claimed. A. R. POWELL.

Production of metal alloys [for tools etc.]. W. SCHROBSDORFF (B.P. 274,866, 19.7.27. Ger., 21.7.26).—A hard alloy for the manufacture of cutting tools comprises a predominating proportion of tantalum, or niobium, or both, a carbide of chromium, molybdenum, tungsten, or uranium, and one or more of the elements cobalt, nickel, copper, palladium, osmium, or iridium. For example, a mixture of 78.2% of tantalum or niobium, 20% of tungsten carbide (W_2C), and 1.8% of cobalt is melted in a vacuum, high-frequency, induction furnace, and the alloy cast into moulds of suitable shape. [Stat. ref.] A. R. POWELL.

Fusible electrodes for use in electric arc-welding. QUASI-ARC CO., LTD., and A. P. STROHMENGER (B.P. 299,575, 15.9.27).—The metallic core is wrapped with matted blue asbestos or similar fibrous mineral silicate containing, if desired, a powder paste or loading consisting of finely-divided iron silicate, ferrosilicon, etc. to form a flux, and the wrapping is cut with helical grooves which are filled in with a cementing material. J. S. G. THOMAS.

Manufacture of electrodes, welding rods, etc. for use in welding and depositing metal. B. TURNER, and FERRO-ARC WELDING CO., LTD. (B.P. 299,163, 24.9.27).—One or both wires to form the twin electrode are coated with a thin film of an adhesive solution of high insulating power, e.g., sodium silicate (cf. B.P. 295,514; B., 1928, 760). J. S. G. THOMAS.

Electrodes etc. for the fusion deposition [arc-welding] of alloys. B. TURNER, and FERRO-ARC WELDING CO., LTD. (B.P. 299,604, 19.10.27).—A welding rod for depositing a nickel-iron or nickel-chromium-iron alloy comprises an iron or steel rod wound with a nickel or nickel-chromium wire spiral, the spaces between the windings of which are packed with a mixture of 200 pts. of powdered chromium, 2.5 pts. of aluminium powder, 35 pts. of fluorspar, 15 pts. of limestone, and 6 pts. of sodium silicate. The whole is coated with a lime-soda-fluorspar flux. The weights of nickel and chromium in the spiral and coating paste are preferably so regulated that the deposited iron alloy has the same composition as "Staybrite" steel. A. R. POWELL.

Manufacture of electrical conductors in light aluminium alloys. COMP. DE PROD. CHIM. ET ELECTRO-MÉTALLURGIQUES ALAIS, FROGES & CAMARGUE (B.P. 286,264, 18.2.28. Fr., 2.3.27).—Age-hardening aluminium alloys are quenched from above 500°, reheated at 125–250° (preferably 150–175°), drawn down to the desired thickness, and re-annealed at 125–250°. The wires have then a high tensile strength combined with maximum electrical conductivity, and are suitable for the manufacture of electrical conductors without a steel core. A. R. POWELL.

Regeneration of chromium-plating baths. E. LIEBREICH (F.P. 627,445, 5.1.27. Ger., 6.1.26).—Chromium-plating baths containing chromic acid, chromates, or more or less reduced hydroxides or oxides of chromium are treated with chromic hydroxide and an alkali compound, preferably an alkali hydroxide. A. R. POWELL.

Electrolytic extraction of aluminium. H. WADE. From ALUMINIUM-IND. A.-G. (B.P. 299,689, 25.10.27. Cf. B.P. 280,881; B., 1928, 644).—To assist the dense deposition of aluminium, small quantities (0.1–0.5%) of manganese, lead, cadmium, nickel, or their compounds or salts are added to the electrolytic bath previously described (*loc. cit.*). Alternatively, these metals may be alloyed with the anode or they may be used as separate auxiliary anodes. M. E. NOTTAGE.

Direct reduction of iron ores. T. LEVOZ, Assr. to N. V. HANDELSMAATSCHAPPIJ FERIRON (U.S.P. 1,689,734, 30.10.28. Appl., 16.2.24. Belg., 1.2.23).—See B.P. 210,795; B., 1925, 765.

Metallurgical product [cast iron]. E. SCHÜZ, Assr. to MEIER & WEICHELT (U.S.P. 1,689,456, 30.10.28. Appl., 3.11.25. Ger., 13.5.24).—See B.P. 234,106; B., 1926, 952.

Spraying and atomising fusible [metallic] material. "METALLISATOR BERLIN A.-G.," and E. GREISER (B.P. 298,858, 27.3.28. Addn. to B.P. 296,546).

Flotation of ores (B.P. 298,736).—See II. **Metal coating for electrodes** (F.P. 620,029).—See XI. **Lead coating of articles** (B.P. 276,643).—See XIII.

XI.—ELECTROTECHNICS.

Separation of emulsions using high-tension alternating currents. S. PAVLIKOVSKI (Przemysl Chem., 1928, 12, 501–525).—The separation of emulsions of sulphuric acid in mineral oil is most efficient when maximum deformation of the electric field is produced. Coagulation of the disperse phase takes place along the lines of force of the field. A certain optimal *P.D.* exists for each apparatus, above which secondary emulsification occurs. R. TRUSZKOWSKI.

Used transformer and switch oils. TYPEK.—See II. **Electrodeposition of chromium.** ROBERTS.—See X.

PATENTS.

Electric furnace. R. B. LINCOLN, Assr. to WESTINGHOUSE ELECTRIC & MANUF. CO. (U.S.P. 1,687,676, 16.10.28. Appl., 16.2.27).—Heating units are arranged along one of the walls of the furnace chamber, and a slot is provided in an adjacent wall to permit the successive removal of the units, the effective length of the slot being less than the combined widths of two of the units arranged below it. J. S. G. THOMAS.

Electric furnace for effecting catalytic gas reactions. G. A. DARZENS (F.P. 625,893, 19.3.26).—The gases or vapours are passed through a vessel packed with small Raschig rings or with a granulated mass which is heated by means of a variable electromagnetic field of high or low tension. The process is suitable for the conversion of alcohol into ethylene. A. R. POWELL.

[Electric induction] furnace lining. J. R. WYATT, Assr. to AJAX METAL CO. (U.S.P. 1,688,220, 16.10.28. Appl., 1.11.23).—Heat-insulating material is rammed in the furnace casing, and an inner lining adapted to hold the molten charge and of low heat-insulating character rests upon the insulator. J. S. G. THOMAS.

Heating liquids by electrical energy. G. BAUM, Assr. to NIAGARA ELECTRO CHEMICAL CO., INC. (U.S.P. 1,688,679–1,688,680, 23.10.28. Appl., [A] 15.10.25, [B] 19.10.26. Austr., [A, B] 28.10.24).—(A) Electric current is passed through an electrically conducting liquid flowing in and not completely filling channels in an electrically non-conducting structure. (B) The orifice of the duct supplying liquid is arranged above non-conducting structures in which small interstitial spaces are arranged between electric current supply mains and exit lines. J. S. G. THOMAS.

Treating liquids with ultra-violet rays. H. SCHOLL (B.P. 283,472, 9.1.28. Ger., 7.1.27).—To prevent the formation of ozone during the irradiation process, air is removed from the liquid, before treatment, by means of a stream of gas, with which the liquid may, if desired, be saturated, the gas becoming chemically active during irradiation. J. S. G. THOMAS.

Gas-filled discharge tubes. N. V. PHILIPS' GLOEILAMPENFABR., Assees. of [A] W. DE GROOT, [B] G. L. HERTZ (Can. P. [A] 265,903 and [B] 266,112, 4.2.25).—(A) The tubes are filled with one or more of the noble gases and with mercury or cadmium vapour; the glass used contains a fluorescent constituent, preferably a uranium compound, and is of a light-diffusing nature. (B) The gas filling consists of argon, krypton, or xenon or of mixtures of these, together with mercury or cadmium vapour. The pressure in the tubes is maintained at 4 mm. A. R. POWELL.

Incandescence cathode for electron-discharge tubes. S. LOEWE (B.P. 274,509, 18.7.27. Ger., 19.7.26).—A core of iron or containing iron is covered with platinum or platinum alloy and coated with metallic oxides or other electron-emitting substances. J. S. G. THOMAS.

Modifying the colour of light in electric discharge tubes. G. CLAUDE and J. M. E. DE BEAUFORT (B.P. 279,053, 21.9.27. Fr., 18.10.26).—Means are provided for readily changing the frequency of the current supply to a discharge tube containing a mixture of gases and metallic vapours. J. S. G. THOMAS.

Vacuum tube. S. G. S. DICKER. From N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 298,963, 16.6.27).—In a vacuum tube built at least partly of quartz a series of glass members having successively increasing coefficients of expansion, *e.g.*, containing aluminium oxide or calcium oxide and no alkali oxide, is sealed to the quartz, for hermetically sealing in the leads. J. S. G. THOMAS.

Neon tube. R. R. MACHLETT, Assr. to RAINBOW LIGHT, INC. (U.S.P. 1,689,146, 23.10.28. Appl., 20.10.26).—Electrodes having an area less than 1 dm.² per ampère of current used are arranged in a tube containing a conducting, monatomic, inert gas, and a mirror of an electropositive metal is deposited on the wall of the tube near the cathode. J. S. G. THOMAS.

Galvanic gas cells. R. VON DALLWITZ-WEGNER (G.P. 450,341, 12.6.24).—The pressure in the gas producer is automatically regulated by means of liquid columns so that the cell is not supplied with more gas than corresponds with the current taken from it, and

at the same time not more gas than the cell can consume is produced in the generator. With hydrochloric acid as electrolyte the cell has an *E.M.F.* about equal to that of the lead accumulator. A. R. POWELL.

Production of insulating and sealing material for electric cells and batteries. E. C. R. MARKS. FROM BATTERIEN- & ELEMENTE-FABR. SYSTEM ZEILER A.-G. (B.P. 299,381, 22.7.27).—Unsaponifiable substances, *e.g.*, paraffin, distillation pitch, are mixed with wax, montan wax, resin, etc., and incompletely saponified with basic substances, *e.g.*, soda lye. After completion of the reaction the material is heated to remove water, and fillers are added. J. S. G. THOMAS.

Metal coating on carbon electrodes for dry batteries. SOC. ANON. HEWITTIC (F.P. 620,029, 9.8.26).—Gas-carbon electrodes are coated by the Schoop spraying process with a layer of nickel-steel containing 40% Ni. The coatings adhere tightly as the coefficient of expansion of the steel is approximately the same as that of the carbon. A. R. POWELL.

Porcelain-like electric insulating substance. J. L. A. HILBRENNER (B.P. 299,408, 26.7.27).—A mixture of clay, kaolin, zirconium oxide, and aluminium oxide, to which, if desired, zinc oxide or a frit composed of magnesium oxide, zinc oxide, and aluminium oxide may be added, is ground in the wet state and quickly sintered. J. S. G. THOMAS.

Galvanic element with carbon and iron electrodes. A. J. B. MAYET (G.P. 450,338, 17.8.26. Fr., 28.7.25).—The electrolyte of a cell with iron and carbon electrodes comprises a solution of hydrochloric acid, ferric chloride, alkali chlorate, and alkali nitrate in such concentrations that a protective film of a basic iron chloride is formed on the negative electrode. The cell has an *E.M.F.* of 1 volt and a low internal resistance. A. R. POWELL.

Contact [current] rectifying device. W. O. SNELLING (U.S.P. 1,686,260, 2.10.28. Appl., 29.7.27).—A finely-divided compound of a metal with an element of the sulphur group is compressed into a coherent mass which is then heated at a temperature sufficient to cause crystal growth without sintering. A. R. POWELL.

Electric coil construction. M. LATOUR, ASSR. to LATOUR CORP. (U.S.P. 1,687,298, 9.10.28. Appl., 17.5.23. Fr., 14.6.22).—The material forming the magnetic circuit of an electrical device is composed of 33-9% Ni, 1-15% Mn, and iron. H. ROYAL-DAWSON.

Incandescence-cathode discharge tube. S. G. S. DICKER. FROM N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 299,098, 22.7.27).

Montan wax (B.P., 299,133).—See II. **Electrolytic brine** (U.S.P. 1,687,433). **Aluminium sulphate** (F.P. 627,334). **Compounds of metal and sulphur** (B.P. 290,986).—See VII. **Porcelain insulators** (F.P. 620,987).—See VIII. **Electric furnace for steel** (U.S.P. 1,687,936). **Electrodes etc. for welding** (B.P. 299,163, 299,575, and 299,604). **Conductors in aluminium alloys** (B.P. 286,264). **Chromium-plating baths** (F.P. 627,445). **Aluminium** (B.P. 299,689).—See X.

XII.—FATS; OILS; WAXES.

Tung oil. IV. Wood oils as crystalloids; "autoxypolymerisation" during drying of wood oils. A. EIBNER and E. ROSSMANN (Chem. Umschau, 1928, 35, 241—250; cf. B., 1928, 825).—The wood oils are fundamentally distinct from other amorphous fatty oils, in that the former consist principally of, or can form, crystallisable glycerides; consequently the earlier colloid theories cannot be applied to these oils. The velocity of isomerisation of α - to β -elaeostearin in tung oil under the influence of light has been studied. Working with thin films of tung oil on glass slides, exposure to sunlight or the light from a mercury-vapour lamp (10 min.) causes rapid formation of crystalline β -elaeostearin; by maintaining the film at 0° precipitation occurs and the crystals can be observed under the microscope by polarised light. At room temperature the β -isomeride is soluble in the bulk of the oil to a considerable extent. Isomerisation did not occur in the absence of light, but tung oil, nevertheless, dried to a clear film when kept in the dark for 3—4 weeks. (Linseed oil was still sticky after 6 weeks.) In the filming (change to amorphous condition) of pure β -elaeostearic acid (m.p. 61°) it was established that oxygen is essential to the process, and that this autoxidation is favoured by light and increase of temperature. An examination of the "autoxypolymerisation" of β -elaeostearin during film-formation shows that the older theory—that drying consisted of a pure polymerisation process catalysed by the presence of oxygen—is invalidated. On exposure of β -elaeostearin to air and light appreciable quantities (7—10%) of oxygen are absorbed during conversion into the amorphous condition; further, examination of the fatty acids recovered from the product indicated that considerable oxidation had occurred. True polymerisation may also take place, and it is submitted that a mixed process ("autoxypolymerisation") proceeds. Thus, if a tung oil containing β -elaeostearin be exposed in a thin film, oxygen is primarily absorbed by the dissolved β -glyceride in the surface layers; the autoxypolymerisation products are at first colloiddally dispersed, and then, by rapid transition to the gel condition, constitute a first disperse phase leading to film formation. Comparative experiments on the behaviour of thin and thick films of tung oil showed that oxygen absorption continues up to the drying point, and decidedly constitutes a principal reaction. It is, however, probable that the whole of the α -elaeostearin in the tung oil does not undergo isomerisation to the β -form with subsequent autoxypolymerisation; the greater part contemporaneously is autoxidised directly to a film in much the same way as the di- α -linolenic linoleic glyceride of linseed oil. E. LEWKOWITSCH.

Tung oil. V. Preliminary cracking and wrinkling of drying wood oil [films]. A. EIBNER and E. ROSSMANN (Chem. Umschau, 1928, 35, 281—290; cf. preceding abstract).—Microscopical examination of films of tung oil drying on an inert base (*e.g.*, glass) shows that the first stage of the drying process consists in the formation of a thin skin, invisible to the eye, which shrinks, producing minute cracks or splits in the film and imparting a cell-like appearance. Drying proceeds with the

absorption of oxygen, and during the process small parallel folds or wrinkles at first appear within the cells at right angles to the boundary cracks, then the pressure exerted by the wrinkling and now expanding film closes the original cracks, thus forcing the edges up into characteristic double ridges. The wrinkling may proceed to such an extent that the straight cell-boundaries become distorted and folded, and finally all cell-like appearance may be destroyed. Similar changes were observed in the drying of β -elæostearin itself. On drying in the dark clear unwrinkled films were obtained; tearing occasionally occurred in thick films in which large amounts of β -elæostearin had been formed by previous irradiation. It is considered that the β -elæostearin is responsible for the characteristic double-ridge structure, by causing the preliminary shrinking and cracking of the film, followed by expansion consequent on autopolymerisation. In brilliant sunlight a clear film may be obtained, the autoxidation of the α -elæostearin proceeding more rapidly than the change to the crack-forming β -isomeride. E. LEWKOWITSCH.

Action of selenium monochloride on fatty oils. E. H. HARVEY and H. A. SCHUETTE (J. Amer. Chem. Soc., 1928, 50, 2837—2840).—The rate of rise in temperature (*a*) and the maximum elevation (*b*) observed when sulphur monochloride or selenium monochloride is added under standard conditions (cf. Fawsitt, A., 1889, 317) to a fatty oil dissolved in xylene are tabulated for a representative series of oils. The values of (*b*) are frequently similar for both reagents, but (*a*) is usually much larger for selenium monochloride than for sulphur monochloride. With the former the heat evolution is the sum of the thermal effects of substitution, addition, and decomposition of the reagent, and is not simply related to either the physical or the chemical properties of the oil. The reaction is, therefore, of no value for the quantitative characterisation of fatty oils.

H. E. F. NOTTON.

Determination of oil in olives. S. KALOYEREA, W. V. CRUICK, and B. E. LESLEY (J. Pharm. Chim., 1928, [viii], 8, 407—415).—The methods are based on the changes in refractive index and density, respectively, of "Halowax oil" (α -chloronaphthalene) after triturating known quantities of the oil and olives together in a mortar. The corresponding constants for the pure olive oil are determined, and from the differences between these and those of the solution of olive oil in "Halowax oil" the amount of olive oil in solution can be determined. Foreign substances from the olive which dissolve can be corrected for as they exert a constant influence. Temperature corrections are also given. A modification is described in which the olives are ground with anhydrous sodium sulphate and then with trichloroethylene, and the density of the resulting solution is determined as above.

E. H. SHARPLES.

Iodine value of Spanish paprika oil. L. C. MITCHELL and S. ALFEND (J. Assoc. Off. Agric. Chem., 1928, 11, 523—527).—The iodine values of the oil extracted from eleven authentic samples of paprika by means of chloroform varied between 133.0 and 139.7; the iodine value range specified by the official stan-

dards (125—136) is therefore not applicable to samples analysed by the chloroform extraction method.

F. R. ENNOS.

Determination of organically-combined sulphur in sulphonated oils. K. H. BAUER (Chem. Umschau, 1928, 35, 290—291).—In reply to criticism levelled by Herbig (Seifensieder-Ztg., 1928, 134), the author points out that, by hydrolysis with *N*-acid, the sulphur is split off from sulphonated oils in the form of the monobasic grouping HSO_4 . Experiments indicate that the results of hydrolysis effected by *N*-hydrochloric and sulphuric acids are influenced by the material of the flasks employed; Jena glass is recommended and blank tests should be performed where possible (cf. B., 1928, 273).

E. LEWKOWITSCH.

Fat in buttermilk. THURSTON and PETERSEN.—See XIX.

PATENTS.

Sulphonation of fats, fatty oils, or fatty acids. H. T. BÖHME A.-G. (B.P. 284,280, 11.1.28. Ger., 27.1.27).—It is claimed that sulphonation products similar to Turkey-red oil, but with an extraordinarily high resistance to acid, lime, and magnesia salts, may be obtained by sulphonating fatty acids or their glycerides in the presence of a large excess of sulphuric acid at temperatures below zero, preferably below -10° . If necessary, the fluidity of the reaction mixture may be increased by the addition of an inert diluent such as a hydrocarbon or its halogenated product. E. HOLMES.

Manufacture of fatty acids containing hydroxy-fatty acids. C. STIEPEL (B.P. 272,198, 27.5.27. Ger., 4.6.26).—Fatty acids containing such proportions of unsaturated hydroxyfatty acids of the ricinoleic acid series that they have approximately the acetyl value and properties of castor oil may be prepared by partially chlorinating products such as Japanese train oil or bone oil, and then heating the resulting mixtures at elevated temperatures with water and caustic alkali. The resulting products have very much higher acetyl values and correspondingly lower iodine values than their original materials. E. HOLMES.

Refining of oils (B.P. 285,064).—See II. **Treating liquids with ultra-violet rays** (B.P. 283,472).—See XI.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Oxidation and weathering of linseed oil paints. D'ANS (Z. angew. Chem., 1928, 41, 1193—1197).—The effect on the oxidation of linseed oil of light and moisture is discussed. The swelling of linseed oil and paint films exposed to moisture, which varies with different pigments, is recommended as a delicate criterion in the evaluation of paints. It is pointed out that, under favourable conditions of warmth and moisture, paint films, especially if recently applied, are susceptible to attack by moulds (*Penicillium*, *Aspergillus*) which may cause rapid and extensive deterioration. The development of moulds appears to be hindered by the presence of zinc oxide and certain coloured pigments: the possibility of attack by moulds should not be neglected when assessing weathering tests. E. LEWKOWITSCH.

Impermanence of many coal-tar colours to lime and zinc white [in paints]. G. ARNOLD (Chem.-Ztg., 1928, 52, 815).—Trials over a period of 12 weeks were made with paints containing, respectively, zinc white, white lead, lithopone, and blanc fixe, with linseed oil and a number of coal-tar dyes (enumerated) on small wood test pieces. Whilst the others were unaffected, the zinc white paints showed the effect of the weather in a few days, and even more rapidly under a glass plate. Similar bad results were given by dyes in conjunction with casein solution and lime.

C. IRWIN.

Testing paints. A. V. BLOM (Z. angew. Chem., 1928, 41, 1178—1181).—For testing the strength of paint films, strips of iron of known tensile strength are painted on one side and then pulled in the usual tensile machine until microscopic cracks begin to appear in the paint film. With freshly painted strips this happens at or near the breaking point of the iron; after the paint has aged somewhat, fracture occurs during the region of plastic flow of the iron, but with very old and brittle films it may occur as soon as the elastic limit is exceeded. The shape and position of the fissures in the paint film after the test provide an indication of the state of preservation of the paint, and, by observing whether the first cracks appear along the Lüder-Hartmann flow lines or whether they are evenly distributed over the surface of the strip, conclusions may be drawn as to the intensity of adherence of aged paint films. The value of the method in testing red lead paint is described; after ageing for 12 days at 80° the elongation of films of ordinary red lead paint decreased from 27 to 16.5%, and that of films of highly dispersed red lead paint from 14.7 to 0.2%. The latter therefore harden and become watertight sooner than the former, and the permanent elongation of 0.2% suffices for paint films on iron as it exceeds the usual limit of proportionality of the metal.

A. R. POWELL.

Determination of wood oil in oil paints. VON REIBNITZ (Farben-Ztg., 1928, 34, 266—268).—Marcusson's method (B., 1926, 500) for the determination of wood oil, depending on its polymerisation with iodine to a solid mass, gives results about 10% low. In the case of paints (without thinners) the extraction of linseed oil with chloroform is difficult and results are frequently high. A better approximation to the truth is obtained if the ascertained value is multiplied by the iodine value of the oil under test, and the product divided by the average of the iodine values of the boiled and unboiled oils. Small quantities of turpentine oil greatly hinder the polymerisation of wood oil and should be removed by shaking with 96% alcohol. Marcusson's method is indirect since the wood oil is determined as the difference between the original oil and the extracted linseed oil. The polymerised wood oil can, however, be saponified with 0.5*N*-alcoholic potash, the fatty acid precipitated with barium chloride, and the excess alkali titrated with hydrochloric acid. This method is described. Comparative tests between it and gravimetric analysis of pure wood oil and mixtures with linseed oil show good agreement. C. IRWIN.

Production of iron [oxide] colours. P. P. BUDNIKOV (Chem.-Ztg., 1928, 52, 846—847).—Magnetic

oxide of iron, produced as a by-product in the manufacture of aniline and α -naphthylamine, is a cheap raw material. After washing, heating for 1 hr. at 500—650°, and grinding, a brown-coloured oxide is produced of poor covering power, but if the residues are treated with 40% of their weight of waste sulphuric acid (70%) and heated for 2 hrs. at 750—800° the colour and covering capacity are greatly improved. The higher the temperature of ignition the darker is the colour. The stronger the acid used the lighter is the colour. Mixtures with gypsum or heavy spar can be used as in the usual process for the production of iron oxide colours.

C. IRWIN.

Nitrocellulose lacquers—some recent developments. S. SMITH (J. Oil & Colour Chem. Assoc., 1928, 11, 342—350).—A general treatment of nitrocellulose lacquers, special reference being made to sources of cellulose, modern solvents of the glycol and other types, and pigments.

S. S. WOOLF.

Lead sulphate. SCHRADER.—See VII.

PATENTS.

Lead coating of articles. H. J. SCHIESS, A. C. BOITEL, E. ZÄHNER, A. BOITEL, and L. SCHIESS [ZÄHNER & SCHIESS & Co.], Assees. of SUBOX A.-G. (ELECTRO CHEM. FABR. METALL- U. HÜTTENPRODUKTE (B.P. 276,643, 2.8.27. Ger., 30.8.26).—Articles may be protected from atmospheric influences by coating them with a paint consisting of a finely-divided tin-antimony-lead alloy in which lead predominates, and a binder, e.g., linseed oil.

M. E. NOTTAGE.

Production of valuable coloured compounds [lakes]. I. G. FARBENIND. A.-G. (B.P. 299,521, 28.7.27. Addn. to B.P. 275,943; B., 1928, 866).—The basic compounds of the prior patent are mixed with basic dyes (or dyes containing both acidic and basic groups) before conversion into phosphotungstates, phosphotungstomolybdates, etc. Examples are: Michler's hydrol with Victoria Pure Blue BO (from tetraethyldiaminobenzo-phenone and ethyl- α -naphthylamine), or with Light Green SF yellowish (green-blue), and *p*-aminobenzaldehyde with Victoria Pure Blue BO (olive-green).

C. HOLLINS.

Preparation of alkyl esters of abietic acid. A. C. JOHNSTON, Assr. to HERCULES POWDER Co. (U.S.P. 1,682,280, 28.8.28. Appl., 29.7.26).—Wood rosin is dissolved in hot aqueous-alcoholic sodium hydroxide, ethyl or methyl sulphate is added, alcohol is distilled off, and the residue is heated at 145°; ethyl or methyl abietate is obtained pure by distillation in a vacuum.

C. HOLLINS.

Manufacture of condensation products of phenols. BRIT. DYESTUFFS CORP., LTD., and E. E. WALKER (B.P. 298,680, 15.7.27).—A phenol is condensed first with an aliphatic ketone and then with an aliphatic aldehyde, one or both condensations being in presence of alkali. A white crystalline product is obtained if after the second condensation acid is added to a pH value of about 6; crystallisation is accelerated by "seeding." The product from phenol, acetone, and formaldehyde has m.p. 123—127°, and may be used for the same purposes as the impure oily product hitherto obtained.

C. HOLLINS.

Preparation of phenol-formaldehyde condensation products. G. PETROV and P. SHESTAKOV (U.S.P. 1,684,142, 11.9.28. Appl., 30.12.25).—A phenol, *e.g.*, cresylic or carboic acid, is condensed with formaldehyde in presence of powdered lead or zinc. Reaction occurs at 75–85°. C. HOLLINS.

Manufacture of artificial materials. SOC. CHEM. IND. IN BASLE (B.P. 283,965, 20.1.28. Switz., 21.1.27).—The working properties and insulating capacity of condensation products such as those made from an aromatic amine and more than 1 mol. of formaldehyde are improved by compressing the porous mass first obtained at elevated temperatures. In an example compression of a mass of *d* 0.6 at 100 atm. and 150° for 1 hr. raised its density to about 1.0. E. HOLMES.

[Paints for] protecting metal, wood, etc. P. W. TURNER (U.S.P. 1,689,567, 30.10.28. Appl., 18.8.26. U.K., 1.12.25).—See B.P. 271,912; B., 1927, 609.

Manufacture of peroxide [ozonides] of hydrocarbons [turpentine substitutes]. A. S. RAMAGE (U.S.P. 1,689,599, 30.10.28. Appl., 27.4.25).—See B.P. 273,832; B., 1927, 661.

Printing ink. K. STEPHAN, ASST. to CHEM. FABR. AUF ACTIEN (VORM. E. SCHERING) (U.S.P. 1,689,368, 30.10.25. Appl., 14.4.27. Ger., 5.5.26).—See B.P. 270,671; B., 1928, 24.

Protective coatings for rubber (B.P. 299,169).—See XIV.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Lamp-black for rubber mixtures. F. KIRCHHOF (Gummi-Ztg., 1928, 43, 309–310).—Chemical analysis can be of distinct value in discriminating between the value of various types of amorphous carbon for rubber-compounding, and examination of a colloid-chemical character is not the only useful procedure. Data are quoted from other workers in support of this opinion. D. F. TWISS.

PATENTS.

Preservative coatings for goods of rubber or like substances. B. D. PORRITT, T. R. DAWSON, and RES. ASSOC. OF BRIT. RUBBER & TYRE MANUFACTURERS (B.P. 299,169, 29.9.27).—Preservative paints or coatings for application to rubber goods for protection against atmospheric influence and the effect of actinic light are made with a medium, preferably capable of swelling the rubber, together with a material, *e.g.*, mineral rubber, to increase viscosity and give some mechanical protection. There are also introduced small quantities of anti-oxidants, *e.g.*, aldol- α -naphthylamine, and of yellow or red substances such as Oil-red S. D. F. TWISS.

Rubber compositions. C. H. CAMPBELL, ASST. to AMER. GLUE CO. (U.S.P. 1,683,862–4, 11.9.28. Appl., 22.10.26).—Reinforcing materials for rubber are obtained by hydrolysing (A) glue, (B) keratin, for 2–6 hrs. with steam at 100 lb. pressure. (C) Both materials are used. C. HOLLINS.

Rubber-like hydrocarbons (B.P. 298,584).—See II. **Rubber-bonded abrasive** (U.S.P. 1,687,410).—See VIII.

XV.—LEATHER; GLUE.

PATENTS.

Manufacturing from gelatin articles such as threads, cords, ribbons, tubes, etc., otherwise made from animal intestinal canals or skins. P. HIRSCH (B.P. 299,105, 25.7.27).—Gelatin solutions, with or without suitable additions, in a thin layer at rest or flowing downwards, and/or the articles made therefrom, are sterilised by exposure to germ-killing rays, preferably ultra-violet rays. D. WOODROFFE.

Manufacture of casein capable of swelling. E. STERN (G.P. 445,732, 6.11.24).—Dry casein is made into a thick paste with a 10% solution of dextrin or a degradation product of starch, and the mixture is dried and ground. A. R. POWELL.

Manufacturing blocks and articles of casein products. A. ZSIGMONDY (B.P. 298,815, 22.12.27).

XVI.—AGRICULTURE.

Colloidal behaviour of soils and soil fertility. V. Distribution of soluble and colloidal iron and aluminium in soils. J. S. JOFFE and H. C. MCLEAN (Soil Sci., 1928, 26, 317–325; cf. B., 1928, 723).—Except in very acid conditions, molecular iron and aluminium compounds do not exist in solution in soils. Examination of dialysed soil extracts shows very little iron or aluminium in the sol form. Most of the iron and aluminium produced during the weathering of complex minerals is distributed throughout the soil in the gel condition. Extraction of soil with barium chloride brings much iron and aluminium into solution, due to liberation of acid following base-exchange effects. The amounts of iron and aluminium so dissolved are less than the equivalent of the acid formed. Less iron than aluminium becomes soluble under these conditions. The amount of iron and aluminium thus appearing in solution may be correlated with the titratable acidity of the acid liberated as a result of base exchange.

A. G. POLLARD.

Influence of nitrogenous fertilisers on the structure of soils. H. LATHER (Z. Pflanz. Düng., 1928, 12A, 227–251).—The structure of soils treated with various fertiliser materials was examined by means of mechanical analyses. The dispersive effect of sodium nitrate on the clay particles was confirmed. On very acid soils and in normal soils where unusually small amounts of nitrate were added a coagulating effect was observed. The results of treating soil with calcium cyanamide were in the main the outcome of its lime content and were somewhat indefinite in the absence of carbon dioxide. In the presence of this latter the usual flocculation occurred. Addition of ammonium sulphate to soil resulted, in the first instance, in interaction with the soil carbonates, and the ammonium carbonate produced greater dispersion of the clay. Subsequent oxidation of the ammonium salt to nitric acid resulted in flocculation of the finer particles. A. G. POLLARD.

Determination of the lime requirement of soils on the basis of laboratory and vegetation experiments. A. KAUFMANN (Z. Pflanz. Düng., 1928, 12A, 156–175).—A comparison was made on five soils of

the Hissink, Gehring, Gedroiz, and Bobko-Askinasi methods. No relationship between the values obtained by these methods was apparent, and it was impossible to say which was the most correct. Pot experiments on these soils were then conducted, using varying amounts of chalk as determined by the titration curve and Daikuhara methods. The amounts as determined by these two methods were insufficient to attain a maximum yield. Gehring's value and a quantity equivalent to three times the value obtained from the maximum yield proved sufficient for a maximum yield. On one soil depression of yield due to over-liming was observed.

H. J. G. HINES.

Detection and determination of chlorate ion in soils. F. MACH and R. HERRMANN (Z. Pflanz. Düng., 1928, 12A, 189—198).—Although the chlorate ion can be detected and determined by treating a soil extract with potassium bromide and potassium iodide and subsequently determining the liberated iodine, the following method is less liable to error. The chlorine ion in a soil extract is first determined by Volhard's method. The chlorate is then reduced by treatment with sulphur dioxide and the chlorine ion is again determined. The amount of chlorate ion is arrived at by difference.

H. J. G. HINES.

Colorimetric process for the determination of the phosphate requirement of arable soils. A. NEMEC (Deut. Landw. Presse, 1928, 55, 296, 331; Bied. Zentr., 1928, 57, 442—443).—Both phosphates and silicates may be determined in soil extracts by the colorimetric method involving the reduction of the respective molybdcic acid compounds with alkaline solutions of quinol. In the examination of cropped soils it is shown that the presence of silicates in soil extracts corresponds with decreased efficiency of phosphatic fertilisers. Soluble silicates favour the re-adsorption or reversion of soluble phosphates in soils. The importance of determinations of soluble silicates in the evaluation of the phosphate requirements of soils is emphasised.

A. G. POLLARD.

Laboratory methods for determining the form in which phosphatic fertilisers should be applied to different soils. L. VON KREYBIG (Z. Pflanz. Düng., 1928, 12A, 176—188).—To suspensions of soil in water are added superphosphate and Rhenania phosphate, respectively. The phosphoric acid in the liquid phase is then determined and a determination of citric-soluble phosphate in the soil is also made. If the superphosphate loses both its water and citric acid solubility, Rhenania or some basic phosphate is likely to give better field results. Similar determinations can be made by the azotobacter method.

H. J. G. HINES.

Fixation of phosphorus by soils. A. G. WEIDEMANN (Soil Sci., 1928, 26, 281—290).—The fixation of phosphorus from monocalcium phosphate solutions by soils under varying conditions is examined. Acid muck soils show a negative fixation with higher concentrations of phosphate solutions. This is assumed to be a consequence of the rendering unfree of a portion of the soil water. Liming prevents this phenomena. Muck soils with relatively high p_H values do not exhibit negative fixation, but their positive fixation capacity is reduced

by acid treatment. If soils showing negative fixation are treated with the amount of water which they can render unfree, phosphate fixation is positive, irrespective of the concentration of the solution used.

A. G. POLLARD.

Use of sulphur and rock phosphate [as fertiliser]. A. KALUSHSKY (Z. Pflanz. Düng., 1928, 12A, 217—226).—In pot experiments with millet rock phosphate was rendered fully available to plants by use with an equal amount of sulphur. Crop yields were as great as when an equal proportion of phosphorus was applied in the form of dicalcium phosphate. The optimum ratio of sulphur to phosphorite varied with soil type, being 4:1 in some soils examined. Sulphur-phosphorite fertiliser produced a considerably increased yield of grain compared with that resulting from an equivalent amount of soluble phosphate.

A. G. POLLARD.

Oxidation of sulphur in suspensions of activated sludge and its influence on the solubilisation of mineral phosphates. C. V. R. AYYAR, T. S. S. PERUMAL, and R. V. NORRIS (J. Indian Inst. Sci., 1928, 11A, 85—90).—Sulphur was rapidly oxidised when introduced into suspensions of activated sludge. The sulphates of manganese, aluminium, and iron (ferrous) acted catalytically in increasing the production of water-soluble phosphates when mineral phosphates were introduced into the system.

H. J. G. HINES.

The "wilting coefficient" and other moisture values in soils. P. I. ANDRIANOV (Z. Pflanz. Düng., 1928, 12A, 145—155).—A simple relationship is demonstrated between the moisture values in soils.

H. J. G. HINES.

Possible relationship between soil salinity and stand in cotton. J. A. HARRIS (J. Agric. Res., 1928, 37, 213—231).—An examination of the possible relationships existing between soil concentration and the seedling stand produced in cultures of three grades of upland cotton grown in experimental fields has given inconclusive results which do not fully confirm those of previous experiments (cf. B., 1926, 842).

E. H. SHARPLES.

Desirable soil-nitrate levels for certain market-garden crops. J. B. SMITH (Soil Sci., 1928, 26, 265—279).—Examination of the nitrate content of soils under crops indicates that the maintenance of a minimum nitrate concentration throughout the growing season is necessary for optimum growth. Desirable concentrations are: for celery, lettuce, and beet 10, cabbage 15, tomatoes and spinach 20 p.p.m. Accumulations of nitrogen following green manuring of soils with oats, rye, and timothy are correlated with the amount of nitrogen in the particular crop used. Oat green manure reduces the relative yield of celery to an extent increasing with the nitrogen content of the green crop. This effect is not attributable to the higher nitrogen accumulation in the soil from the decomposed oat crop.

A. G. POLLARD.

Toxic fluorine compounds. MARCOVITCH.—See VII.

PATENTS.

Treatment of plants. W. EGGERT, JUN. (U.S.P. 1,686,964, 9.10.28. Appl., 22.12.24).—Tobacco plants are treated in the blossoming season with a solution

obtained by the fermentation of leaves and petals of the same type of plants, together with a sweetening substance and a derivative of gum benzoin.

A. R. POWELL.

Treatment of seed. A. KLAGES (U.S.P. 1,688,720, 23.10.28. Appl., 17.5.26. Ger., 27.10.23).—See B.P. 274,974; B., 1927, 792.

XVII.—SUGARS; STARCHES; GUMS.

Influence of the basicity of basic lead acetate in the clarification of molasses solutions. E. SOMMER (Z. Zuckerind. Czechoslov., 1928, 53, 45—52).—The basic lead acetate prescribed for sugar analysis contains 31.9% of its lead in the form of oxide. If solutions containing a higher proportion of oxide to normal acetate are used, lower values are obtained for the polarisation of beet molasses, assuming that the same total amount of lead is used for a given weight of molasses. If the defecant contains 57% of its lead as oxide it may lower a molasses polarisation of 50 by 1—2 units. The extent of the lowering is a direct function of the original alkalinity (p_H value) of the molasses; but no alkalinity likely to occur in practice will produce any lowering when basic lead acetate of the usual composition is employed. The decolorising effect (always assuming the use of the same total amount of lead for a given weight of molasses) increases with the proportion of lead oxide until this reaches 39—52%, but beyond this limit it diminishes rather rapidly. With 48% of the total lead as oxide decolorisation is much better than with the usual proportion, and the lowering of polarisation can be prevented by previous neutralisation of the solution with acetic acid.

J. H. LANE.

Action of basic lead acetate in presence of alkali on dextrans and gums, and its use in the analysis of certain sugar products. C. PERTUSI (Annali Chim. Appl., 1928, 18, 414—420).—Dextrans and gums are precipitated by basic lead acetate in presence of sodium hydroxide, and the preparation of the solution in which the sugars present in biscuits may be determined may be carried out as follows: 20 g. of the material, made into a paste with a little water, are introduced into a 200—220 c.c. Mohr flask and mixed with about 150 c.c. of water. After some hours the mass is treated with 5—6 c.c. of basic lead acetate solution, two or three drops of alcoholic phenolphthalein solution, and, drop by drop, concentrated sodium hydroxide solution until a faint pink colour appears. After some time the liquid is made up to volume and filtered, any slight coloration in the filtrate being usually removable by addition of a drop of acetic acid solution. When sucrose is the only sugar present this method gives good results, in agreement with those furnished by Savini's method (cf. B., 1917, 96). When invert sugar also is present and Clerget's method for determining the sucrose has to be employed, the results, as is also the case with Savini's method, are not so good; determination of the invert sugar is also not so satisfactory. With gum products 20 g. are treated with about 150 c.c. of hot water in a Mohr flask, and 20 c.c. of basic lead acetate solution are added in quantities alternately with sodium hydroxide. When the precipitate collects

into a consistent mass the precipitation of the gum is practically complete. The subsequent procedure is as above.

T. H. POPE.

Significance and determination of temperature coefficients in the study of inversion. V. ZAFOUK (Z. Zuckerind. Czechoslov., 1928, 53, 73—79).—Measurements of the inverting capacity of weak acids at different p_H values showed that no inversion occurs beyond about p_H 6. The p_H values of solutions of weak acids, or of mixtures of the acids and their salts, vary to some extent with temperature, and the variations are not the same with different acids; differences up to 0.1 in p_H value were observed between 20° and 70°. Curves are given showing the p_H values and corresponding temperature coefficients for different stages of neutralisation of succinic, aspartic, acetic, glutimic, and glutamic acids. For practical purposes a factory juice which is slightly alkaline to phenolphthalein will be safe from inversion, provided it has no tendency to become more acid in course of time or on heating.

J. H. LANE.

Determination of sugars from the oxygen in the cupric oxide. M. D. HADJIEFF (Z. Unters. Lebensm., 1928, 55, 613—614).—The cuprous oxide is precipitated by the Meissl-Allihn method and collected in an unweighed Allihn asbestos filter tube. It is dried at 120—130° and oxidised by heating in a stream of dry air, and the tube and its contents are cooled in a desiccator and weighed. The cupric oxide is then reduced to copper in a stream of pure dry hydrogen, cooled in the gas, and weighed again. The difference in the two weighings multiplied by 3.9731 gives the weight of copper, from which the corresponding weight of dextrose can be found in the usual way. Concordant results are obtained agreeing well with those obtained by the usual Meissl-Allihn method.

W. J. BOYD.

Polarimetric determination of sucrose in mixtures of milk and sucrose. G. W. MONIER-WILLIAMS (Analyst, 1928, 53, 569—581).—About 350 g. of the milk and sucrose mixture (weighed to 0.1 g.) are treated with 1.5 g. of (powdered) citric acid, the coagulated milk is shaken, and to it is added a mixture of 9 g. of phosphotungstic acid and 45 g. of dry sand, gradually and with shaking. A determination of the total solids is carried out on the filtrate. Then 50 c.c. of filtrate are weighed into a 100 c.c. flask, the amount of water is calculated from the total solids determination (y in formula), 2.675 g. of dry ammonium chloride are added, and the liquid is made up to volume. After 1 hr. the solution is polarised in a 200 mm. water-jacketed tube at 18—22°. A second portion of the filtrate is measured into the same flask with the same pipette, and 10 c.c. of 5*N*-hydrochloric acid are added, the mixture being placed in a water-bath at 60° for 12 min. with shaking for the first 4 min. On cooling, 10 c.c. of 5*N*-ammonia solution are slowly added to neutralise the hydrochloric acid present, the solution is made up to volume, filtered if necessary, and polarised in the same way and as nearly as possible at the same temperature as the uninverted solution. The mean of the readings is B , and $A-B$ the change of rotation on inversion. Then % sucrose = $(A-B)/[87.9+0.06c-0.3(t-20)] \times 100 \times 2y$, where c is the % concentration

of total sugars in the inverted solution as diluted for polarisation, t the temperature of invert polarisation, x the % of water by wt. in the diluted condensed milk or milk and sucrose mixture, and y the weight of water in the 50 c.c. of filtrate taken for polarisation. Notes on the method have reference to the temperature of inversion; the strength of hydrochloric acid and time of inversion; the neutralisation of inverted solution and addition of ammonium chloride to the direct rotation liquid; the divisor in the formula; the total solids determination in milk and sucrose filtrates; the ratio correction; and to a method for maintaining a constant temperature in the polarimeter.

D. G. HEWER.

Sulphide-stain method for determining small quantities of "volatile sulphur" in sugar. J. M. BRYAN (Analyst, 1928, 53, 589—590).—In a modification of Mann's sulphide-stain method (cf. Ogilvie, B., 1927, 312) a Gutzeit apparatus is used, and lead acetate test papers (6 cm. \times 4 mm.). The sugar sample is added to the flask containing 25 g. of zinc pellets (washed with dilute hydrochloric acid and air-free distilled water) and 50 c.c. of air-free distilled water; 50 c.c. of 1:1 hydrochloric acid are added, the contents are shaken, and, after 1 hr., the test paper is dipped in melted paraffin and compared with the standard stains which correspond to 0.005, 0.010, 0.015, 0.020, and 0.025 mg. of sulphur dioxide when 5 c.c. of the solutions and 10 g. of pure sucrose per 100 c.c. are used in the apparatus. A blank test must be made, and a preliminary trial. Comparison showed that 1 pt. of hydrogen sulphide was approximately equivalent colorimetrically to 2 pts. of sulphur dioxide.

D. G. HEWER.

Analysis of starch sugar degradation products by selective fermentation. T. McLACHLAN (Analyst, 1928, 53, 583—588).—The method found most satisfactory for analysing starch degradation products was selective fermentation by different yeasts. As a general routine method, 8 tubes each containing 50 c.c. of a 10% solution of the material being examined are steam-sterilised for three successive days with rapid heating on the first day to destroy any diastase present. Two tubes are inoculated with *Saccharomyces exiguus*, two with *S. Froberg*, two with *S. Saaz*, and two are kept blank, and the set is incubated at 26° for 14 days, the tubes being rotated on the 4th or 5th day to distribute the yeasts and introduce a fresh air supply. One tube from each yeast is then emptied into a separate 150 c.c. beaker, evaporated to about 15 c.c., cooled, made up to 50 c.c., and the gravity determined, the second set of tubes being kept for verification. The difference between the total solids of the blank and of the solution fermented by *S. exiguus* represents dextrose and laevulose, that between *S. exiguus* and *S. Froberg* represents maltose, and that between *S. Froberg* and *S. Saaz* gives other fermentable sugars. For determination of dextrins, the optical rotation is determined and calculated for 100% product; then % dextrins = $[\alpha]_D \times 100/180$. It is suggested that values should always be calculated on 100% total solids. Analyses are given of four samples of commercial glucose and twelve of malt extracts.

D. G. HEWER.

Continuous automatic counter-current extractor for starchy and saccharine substances with press elevator. G. MEZZADROLI and G. MAZZINI (Zymologica, 1928, 3, 109—112).—Four or six superposed, steam-jacketed, horizontal cylinders are provided with screws which are driven from the outside and have tubular axes for steam heating. The material, such as beet slices, is fed into the uppermost cylinder through a charger with hermetic seal and passes from end to end of each cylinder in turn, water, previously pumped through a heater, traversing the cylinders in the opposite direction to be discharged through a grid which is kept clear by the screw moving against it. The exhausted material is carried by a screw elevator against a pressure cone and so freed from most of the absorbed liquid. The apparatus is simpler than the diffusion battery, and requires less space and less labour, whilst the heat is more efficiently utilised and initial and maintenance costs are diminished.

T. H. POPE.

PATENTS.

Cyclic process of using barium compounds in the manufacture of sugar. H. W. DAHLBERG (U.S.P. 1,688,071, 16.10.28. Appl., 16.22.25).—Insoluble material containing barium carbonate is heated with alumina thereby producing a barium aluminate compound containing water-soluble barytic material, which is used in the treatment of molasses. The insoluble material recovered from the barium saccharate is heated again with alumina to produce water-soluble barytic material.

W. J. BOYD.

Manufacture of sugar [from molasses]. T. GASPAR Y ARNAL (B.P. 297,482, 23.5.27).—Molasses is prepared for desaccharification by treatment with soluble ferrocyanides, with or without an alcohol, to precipitate alkali and alkaline-earth metals as double ferrocyanides. (Cf. B., 1928, 725.)

J. H. LANE.

Simultaneously making sugar and paper pulp from [sugar] cane. E. A. VAZQUEZ, ASST. to VAZCANE PROCESS, INC. (U.S.P. 1,688,904—5, 23.10.28. Appl., [A] 30.8.27, [B] 3.4.28. Cuba, [A, B] 2.6.27).—Sugar cane is simultaneously extracted and reduced to fibre by contact with a moving abrasive surface in the presence of extracting liquid, and, after separation from the fibres, the solution is worked up to sugar.

L. A. COLES.

Recovering sugar values. R. C. CAMPBELL, ASST. to UNITED FILTERS CORP. (U.S.P. 1,685,118, 25.9.28. Appl., 7.5.25).—The mud is filtered and washed continuously, the wash water being collected and the cloudy filtrates returned. After each cycle of operations the filter surfaces are cleaned.

F. G. CLARKE.

Increasing the stability of solid sugar, more particularly starch sugar. H. MAUTNER (B.P. 297,338, 15.6.27).—To prevent fermentation consequent on absorption of moisture, dextrose, or other sugar is incorporated with a small proportion of alcohol, not sufficient to render it wet. E.g., alcohol may be added to a very concentrated dextrose solution, which is afterwards allowed to crystallise to a solid mass.

J. H. LANE.

Clarification of cane molasses for the growing of yeast. C. HOFFMAN, C. N. FREY, and F. M. HILDE-

BRANT, ASSTN. to FLEISCHMANN CO. (U.S.P. 1,687,561, 16.10.28. Appl., 8.5.23).—Sufficient sodium silicate is added to a dilute solution of molasses to give p_H 7.7—12. The liquid is then filtered while hot.

W. J. BOYD.

Treatment of molasses [for use in manufacture of yeast]. F. RANSOHOFF, ASST. to FLEISCHMANN CO. (U.S.P. 1,688,831, 23.10.28. Appl., 11.5.25).—The molasses is made alkaline with ammonia, an ammonium phosphate is added, and insoluble substances are separated.

W. J. BOYD.

[Cooling] treatment of sugar and like material. A. E. WHITE. FROM ACKERS, LONSDALE & CO. PROPRIETARY, LTD., and G. LONSDALE (B.P. 298,735, 18.8.27).

XVIII.—FERMENTATION INDUSTRIES.

Preservative principles of hops. IX. Influence of special methods of drying at low temperatures on the antiseptic properties of hops. J. J. H. HASTINGS and T. K. WALKER (J. Inst. Brew., 1928, 34, 556—565).—Hops dried under special conditions at a low temperature have an initially greater antiseptic power than those subjected to the ordinary kiln processes of drying. This additional antiseptic power is lost after six months in cold storage, and the specially dried and kiln-dried hops then possess approximately equal antiseptic values. The extra antiseptic power of green and specially dried hops cannot be ascribed to the presence of any hitherto unidentified antiseptic substance, which is destroyed by kiln drying, but is due to the presence of a large proportion of crystalline lupulon in the β -portion of the soft resin. This is partly destroyed when the hops are dried by ordinary kiln processes. Humulon and, to a greater degree, lupulon are progressively destroyed when hops are boiled in water or wort. The actual proportion of the two components destroyed depends chiefly on the time factor.

C. RANKEN.

Brewing trials with new and commercial varieties of hops. W. T. SMITH and A. J. C. COSBIE (J. Inst. Brew., 1928, 34, 565—570).—The brewing value, flavour, and preservative property of certain of the new varieties of hops raised at Wye are compared with well-known commercial varieties. Among the tabulated results of the trials a hop of Oregon and English parentage takes a high place. Retaining only to a slight extent the rank Oregon aroma of its female parent, it is superior to English hops in preservative power, and gave a beer of exceptionally high class and distinctly aromatic. In all cases hops affected with Downy Mildew imparted their undesirable flavour to the beer in which they were used.

C. RANKEN.

Small-scale brewing in the laboratory. F. E. DAY (J. Inst. Brew., 1928, 34, 570—573).—Brewing conditions are imitated as closely as possible, the duration of each operation being considered as important as the temperature or the proportion of the ingredients. The main difficulty of controlling temperatures during fermentation is overcome by the use of vacuum flasks as fermenting vessels which allows the temperature during fermentation to rise slowly from 15° to 20.5°, and at the end to fall to the original figure without sudden cooling.

C. RANKEN.

Determination of glycerol in beer. H. LACROIX and S. KROPACSY (Woch. Brau., 1928, 45, 490—491).—Zeisel and Fanto's method is applicable to beers, but certain minor modifications are advisable. Duplicate results on dark beers vary more than those on pale beers, but in any case the method is more rapid and gives better results than the lime method.

F. E. DAY.

Action of chloroform on development of yeast. M. N. MEISSEL (Woch. Brau., 1928, 45, 488—490).—Yeast cultures of various ages were spread on the surface of wort-agar in Koch dishes and exposed to the action of chloroform vapour. On removal the yeasts showed no development for 3—7 days, against 1 day for the controls. Young cultures were more affected than old, both as regards narcosis and death of the cells. From a pure culture of *Saccharomyces cerevisiae* so treated for a considerable period, cells survived which showed a permanent change in cultural characters, being asporogenous and forming intensely wrinkled colonies.

F. E. DAY.

Balsam vinegar of Modena. E. PARISI (Annali Chim. Appl., 1928, 18, 395—407).—This vinegar, which, to be of good quality should be at least 100 years old, is made by storing a mixture of boiled, concentrated must, usually of the white Trebbiano grape, with strong vinegar in casks of oak, chestnut, or mulberry wood. It is an almost syrupy, black liquid of sweet, acid taste and pleasant odour, and it emits vapours which produce stimulating effects when breathed. It is not used alone, but is mixed with good ordinary vinegar. Analysis of eight samples, five dating back to 1800—1860 and the others of unknown age, gave: d_{20}^{20} 1.1055—1.2880; extract 23.41—79.77 g. per 100 c.c.; fixed acid 2.77—4.42, and volatile acid 3.27—13.78, calculated as g. of acetic acid per 100 c.c.; reducing sugars 26.28—49.44 g. per 100 c.c.; glycerol 0.67—0.91 g. per 100 c.c. Among the components of these vinegars are acetyl-methylcarbinol, acetaldehyde, alcohol, formic and acetic acids, much malic, less succinic, and little tartaric acid, and esters. A moderately good balsam vinegar is now made by adding old vinegar of good quality to boiled must.

T. H. POPE.

Analysis of starch sugar. McLACHLAN.—See XVII.

PATENTS.

Production of nitrites (U.S.P. 1,685,629).—See VII. **Molasses for yeast** (U.S.P. 1,687,561 and 1,688,831).—See XVII. **Curing meat** (U.S.P. 1,685,630).—See XIX.

XIX.—FOODS.

Behaviour of bacteria in milk. G. DEI GRISOGONO (Zymologica, 1928, 3, 113—115).—The existence of Gorini's two types of coagulation of milk (A., 1926, 1278; B., 1926, 418) is confirmed. Organisms causing the first type of coagulation do not liquefy gelatin, whereas those producing the second type liquefy gelatin distinctly and rapidly. The only strain of streptococcus isolated from milk belongs to the second class.

T. H. POPE.

Detection of added water in sour milk by determination of the refractive index. A. SCHNECK (Milch. Zentr., 1928, 57, 309—312).—During the souring of a milk the refractive index of its calcium chloride serum increases, but the specific refraction remains constant. The increase in the dry substance on souring is a linear function of the acidity, from which it may be calculated. The total dry substance of the serum may be obtained from the refraction by means of Wiegner's table, and hence the amount of dry substance and the refraction of the serum of the original milk are known. In the case of a sour milk derived from milk adulterated with water, the calculated dry substance and refraction are below the normal. F. R. ENNOS.

Lipins and sterols as sources of error in the determination of fat in buttermilk by ether extraction methods. L. M. THURSTON and W. E. PETERSEN (J. Dairy Sci., 1928, 11, 270—283).—For a product such as buttermilk, containing approximately as much lecithin as fat, Babcock's method for the determination of fat is preferred. The gravimetric method gives high results owing to the extraction of unsaponifiable matter and lecithin. CHEMICAL ABSTRACTS.

Soluble carbohydrates in rye flour. T. CHRZASZCZ and W. MICHALSKI (Przemysl Chem., 1928, 12, 389—402).—Rye flour contains normally as much dextrin as reducing sugar. After storage the dextrin content is trebled, whilst where unsuitable damp magazines are used the sugar content rises at the expense of the dextrin. Better yields of bread are obtained from brown than from white flours. The baking qualities of brown flour improve up to 1½ years of storage, but after 3 years a distinct deterioration may be observed. The temperature of milling should not rise above 40°, as otherwise destruction of diastase contained in the wheat kernel takes place. R. TRUSZKOWSKI.

Evaluation of flour by iodine solution. T. CHRZASZCZ and W. MICHALSKI (Przemysl Chem., 1928, 12, 342—349).—The quantity of iodine adsorbed in unit time by an aqueous extract of flour is in proportion to the dextrin and maltose content of the latter. Since the content of these substances depends on the condition of the grain at harvest and on the milling and storage of the flour, this method affords a convenient means of evaluating it for breadmaking. R. TRUSZKOWSKI.

Vitamin contents of Japanese foodstuffs. SHIMODA, FUJIMAKI, and S. SAIKI (Imp. Japanese Inst. Nutrition; Bull. soc. hyg. aliment., 1927, 15, 481—504, 524—551).—The following were studied: summer oranges, canned plums, lard, radish juice, *Tapes Philippinarum*, radish leaves, fermented rice, preserved radish, cucumber, and egg-plant, buckwheat flour, red kidney beans, and Japanese eels. CHEMICAL ABSTRACTS.

Proteins of Indian foodstuffs. I. Proteins of ragi (*Eleusine Coracana*): eleusin, the alcohol-soluble protein. N. NARAYANA and R. V. NORRIS (J. Indian Inst. Sci., 1928, 11A, 91—95).—An improved method of extraction consists in pouring the alcohol solution into water and subsequent precipitation by an electrolyte. A preliminary examination by the Van Slyke method indicates that eleusin is characterised by its low content of basic nitrogen. H. J. G. HINES.

Influence of malic acid in lemon juice on Warington's method of analysis. F. PERCIABOSCO (Atti II Cong. Naz. Chim. Pura Appl., 1926, 1369—1370; Chem. Zentr., 1928, i, 2191).—Addition of 10% of malic acid to lemon juice does not affect the determination of citric acid by Warington's method, calcium malate being ten times as soluble as the citrate. A. A. ELDRIDGE.

Determination of phosphoric acid in foodstuffs. W. HARTMANN (Z. Unters. Lebensm., 1928, 55, 610—613).—The following solutions are prepared: (a) 210 g. of ammonium molybdate in 300 c.c. of water and 400 c.c. of 10% ammonia solution, (b) nitric acid (d 1.2), (c) 1% solution of potassium nitrate. Solutions (a) and (b) are mixed shortly before use in the proportions 1:2 and 75 c.c. of this mixture are added to the phosphate solution in the cold, 0.5 g. of ammonium sulphate having been previously dissolved in the latter. The mixture is stirred with a glass rod until precipitation occurs, without touching the sides of the beaker. After about 4 hrs. it is filtered through a close "barium sulphate" paper and the precipitate is washed with 1% potassium nitrate solution until 10 c.c. of the wash liquor are neutralised to methyl-orange by 1—2 drops of 0.1N-soda. The filter and precipitate are placed in a beaker, covered with 1% potassium nitrate solution, and the precipitate is dissolved by gradual addition of carbonate-free N-soda. The excess of soda is determined by titration with N-sulphuric acid. Satisfactory results were obtained for a standard phosphate solution, and determinations of phosphoric acid in wine and of alcohol-soluble phosphoric acid in dried eggs gave results in agreement with those obtained by the magnesium pyrophosphate method. W. J. BOYD.

Determination of cacao shell. W. PLÜCKER, A. STEINRUCK, and F. STARCK (Z. Unters. Lebensm., 1928, 55, 622—623).—A question of priority (cf. B., 1928, 314). W. J. BOYD.

Determination of sucrose in mixtures of milk and sucrose. MONIER-WILLIAMS.—See XVII.

PATENTS.

Increasing the percentage of fat in milk, and production of artificial creams. H. BÜNING (B.P. 299,617, 7.11.27).—Earth-nut oil, with or without addition of sesamé oil, is hardened to 32° and added to milk in the proportion of 10 pts. of oil to 90 pts. of milk. The mixture is homogenised at 175 atm. for enriched milk, at 150 atm. for coffee-cream, and at 50—75 atm. for whipped cream. In producing the last-named product the milk must not be pasteurised prior to addition of the oil, and should preferably be unskimmed. The homogenised mixture is skimmed and the cream brought to a fat content of 35%. By using a higher proportion of oil, a cream containing 35% of fat may be obtained directly. W. J. BOYD.

Production of chocolate-milk compositions. W. A. HEYMAN (U.S.P. 1,689,028—9, 23.10.28. Appl. 4.5.27).—(A) Cocoa powder of low fat content is mixed with a suitable liquid, the mixture is heated to break up the starch cells, homogenised, and mixed with a lactil fluid. The resultant mixture is sterilised or pasteurised by heat and dried to a powder. (B) A soluble powder is

produced consisting of acid-neutralised cocoa of low fat content with the starch cells broken up, together with milk solids.

W. J. BOYD.

Production of egg products. A. K. EPSTEIN (U.S.P. 1,687,268—1,687,270, 9.10.28. Appl., 24.12.26).—(A) Egg yolk is mixed with an edible substance which forms hydrogen ions, and is then frozen. Sufficient acid-producing substance is added to reduce bacterial decomposition before freezing and during thawing, and to modify the physical condition of the thawed product. (B) A neutral, soluble, edible salt is also added to control the viscosity of the thawed product. (C) A mixture containing egg yolk, a neutral organic compound having at least one hydroxyl group and capable of lowering the freezing point, *e.g.*, sucrose, and a neutral salt, *e.g.*, sodium chloride, is frozen; an edible acid may also be present. The addition of sodium chloride and of the organic compound reduces the viscosity of the thawed acid-treated yolk; a neutral salt added to a mixture of the yolk and organic compound increases the viscosity of the thawed product.

F. G. CLARKE.

Curing meat. E. T. DRAKE, ASST. to CUDAHY PACKING Co. (U.S.P. 1,685,630, 25.9.28. Appl., 8.2.26).—Meat is immersed in a pickle prepared by inoculating an aqueous solution of a nitrate, sugar, salt, and a protein material with a selected group of nitrate-reducing and salt-tolerant bacilli or spirilli.

C. RANKEN.

Preparing spinach and the like for canning. W. E. THOMAS (U.S.P. 1,685,511, 25.9.28. Appl., 27.9.27).—The vegetable is wilted at the maximum temperature at which the formation of phaeophytin from the chlorophyll present would be insufficient to affect the natural colour of the vegetable.

H. ROYAL-DAWSON.

Manufacture of phosphorus compound from animal proteids. S. POSTERNAK, ASST. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,690,752, 6.11.28. Appl., 21.3.27. Switz., 31.3.26).—See B.P. 268,805; B., 1928, 138.

Manufacture of chocolate goods and the like. R. HEAD, ANC. ÉTABL. A. SAVY JEANJEAN & CIE. SOC. ANON., and BAKER PERKINS, LTD. (B.P. 299,340, 22.7.27).

Manufacture of compressed edible tablets. R. SUCZEK (B.P. 299,685, 18.5.28).

Treating liquids with ultra-violet rays (B.P. 283,472).—See XI.

XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Detection of "plasmoquin." W. SCHULEMAN, F. SCHÖNHÖFER, and A. WINGLER (Abh. Gebiete Auslandskunde, 1928, 26, D, ii, 5 pp.; Chem. Zentr., 1928, i, 2193).—"Plasmoquin" is detected in urine-free aqueous solution at a dilution of 1 in 2×10^5 by the development of a blue colour with tetrachlorobenzoquinone. Reaction with potassium mercuri-iodide, which is not specific, is sensitive to 1 in 10^5 .

A. A. ELDRIDGE.

Formula for calculating composition of mixtures of mydriatic alkaloids. J. C. MUNCH and G. S. GITTINGER (J. Assoc. Off. Agric. Chem., 1928, 11, 521—523).—The physiological effect of a mixture of

mydriatic alkaloids is the sum of that of each of the constituents; from this a formula is derived for the composition of a two-fold mixture when the total alkaloid present and the relative activity of the constituents are known.

F. R. ENNOS.

PATENTS.

Production of primary stibinic acids. F. DUNNING and E. E. REID, ASSRS. of HYNSON, WESTCOTT, and DUNNING (U.S.P. 1,682,269, 28.8.28. Appl., 6.1.27).—*p*-Aminoacetanilide and antimonious oxide are dissolved in hydrochloric acid and diazotised. The diazo-antimony compound is suspended in water and added to 10% sodium hydroxide solution at 90°. On acidification *p*-aminobenzenestibinic acid is precipitated.

C. HOLLINS.

Manufacture of water-soluble anthraquinone glucosides. A. CARPMAEL. From I. G. FARBERIND. A.-G. (B.P. 298,674, 14.7.27).—Extracts of drugs containing anthraquinone glucosides (cascara sagrada, frangula), made, *e.g.*, by extraction of the drug with alcohol, evaporation, and dissolution of the residue in water, are purified by treatment with metallic hydroxides (*e.g.*, of aluminium, manganese, iron). On evaporation, pure mixtures of anthraquinone glucosides are obtained.

B. FULLMAN.

Process for obtaining alkaloids. H. A. GILL. From N. V. CHEM. FABR. "DELTA" (B.P. 298,787, 4.11.27).—The finely-divided parts of the plants containing alkaloid are reduced to a thin pulp with water, milk of lime, etc., and the pulp is treated, in counter-current, in a column with steam, which removes the alkaloids volatile in steam.

B. FULLMAN.

Process for obtaining alkaloids. G. W. F. F. KNOTH (U.S.P. 1,686,866, 9.10.28. Appl., 17.11.27. Ger., 11.10.26).—An aqueous alkaline pulp of the finely-divided plant is passed down a column, and the alkaloid is removed by an upward current of steam.

F. G. CLARKE.

Refining of tobacco. ZIGARETTENFABR. LESMONA GES.M.B.H., and E. ROSENHOCH (B.P. 293,760, 10.7.28. Ger., 11.7.27).—The rib ends of the leaves are brushed to remove insects, larvæ, etc., and moistened slightly with sterilised water; after several hours the leaves are separated from one another, treated to remove foreign matter, moistened all over with sterilised water, and, after 10—16 hrs., treated in a conveying apparatus with a counter-current of ozone, the time of treatment and speed of the ozone current increasing with increasing nicotine content.

L. A. COLES.

Manufacture of physiologically-active substances from ovaries, corpus luteum, and placenta. M. HARTMANN, ASST. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,690,932, 6.11.28. Appl., 14.4.24. Switz., 8.5.23).—See B.P. 226,372; B., 1925, 149.

Manufacture of derivatives of quinolinecarboxylic acids. K. MIESCHER, ASST. to SOC. CHEM. IND. IN BASLE (U.S.P. 1,688,469, 23.10.28. Appl., 19.4.27. Switz., 30.4.26).—See B.P. 270,339; B., 1928, 730.

Refining of oils (B.P. 285,064).—See II.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Law of blackening of the photographic plate at low densities. E. A. BAKER (Proc. Roy. Soc. Edin., 1928, 48, 106—133; cf. B., 1925, 569; 1927, 461).—A theory is developed in which each grain of a photographic emulsion is assumed to act as an absorbing and emitting system, the rate of development of the grain depending on the particular sequence of absorptions and emissions which it has undergone. Comparison of the theoretical results obtained when the proportion of grains which suffer given sequences of absorptions and emissions is small, with experimental determinations of the variation of density with exposure time, indicates that the grain requires two successive absorptions or three absorptions with one intermediate emission to render it susceptible respectively to "full" or "partial" development. The development is "partial" when it is stopped at an early stage so that the grains developed are selected from those belonging to a small group of states of very similar properties. Development sufficient to blacken grains which have been reversed by short exposure to intense light is termed "full." The results apply over the whole range of wave-length tested, from 4800 Å. to below 2000 Å. A quantitative theory of the action gives good agreement with the facts relating to pre-exposure, inefficiency of short exposure to intense light, proportions of grains affected by different exposures in single-layer plates (for small grains), and influence of development on Schwarzschild's index. In the case of the "curves of constant blackening" there is a disagreement between theory and experiment for long exposures, which indicates that the "emission," though spontaneous, does not obey the unimolecular law. A new formula for the characteristic curve of a plate with uniform grains, successful over the low and moderate density region, is derived. A. B. MANNING.

PATENTS.

Varying the tones of photographic diazo prints. KALLÉ & Co. A.-G. (B.P. 287,063, 12.3.28. Ger., 11.3.27. Addn. to B.P. 280,593; B., 1928, 549).—Diazo prints made with the aid of titanium salts by the process of the prior patent are toned by exposure to moisture; e.g., the yellowish-browns obtained with 1:2:4-diazonaphtholsulphonic acid, dichlororesorcinol, and potassium titanium oxalate become dark brown by treatment with water vapour, moist air, or a damp sponge. C. HOLLINS.

Production of photo-prints and photo-copies. I. G. FARBENIND. A.-G. (B.P. 289,386, 25.4.28. Ger., 25.4.27. Addn. to B.P. 286,233; B., 1928, 466).—In the process of the prior patent (cf. also B.P. 286,736; B., 1928, 549) a salt of a substituted β -naphthaquinone-4-sulphonic acid is used as the light-sensitive substance, e.g., the 6-chloro- or 6-sulpho-derivative. C. HOLLINS.

XXII.—EXPLOSIVES; MATCHES.

PATENT.

Dynamite composition. N. G. JOHNSON and S. G. BAKER, JUN., Assrs. to E. I. DU PONT DE NEMOURS & Co.

(U.S.P. 1,687,023, 9.10.28. Appl., 18.7.27).—A liquid explosive is mixed with ground popped corn; the composition has a low density. F. G. CLARKE.

XXIII.—SANITATION; WATER PURIFICATION.

"Natrolite" filter for water-softening. S. TYDÉN (Svensk Kem. Tidskr., 1928, 40, 250—256).—The water-softening properties of a filter of the zeolite type are studied. Analysis of the filtering medium showed it to consist mainly of the silicates of aluminium and sodium, corresponding to a formula $RO, 2R'_2O_3, 9SiO_2$, where $R = Ca, Mg, K_2, Na_2$ and $R' = Fe, Al$; about 1% of titania was also present. Experiments with water of hardness 9 showed that this value could be reduced to zero by passage of the water through the filter; regeneration was carried out by means of salt solution in the usual way. H. F. HARWOOD.

Toxic fluorine compounds. MARCOVITCH.—See VII. **Sulphur-oxidation of activated sludge.** AYAR and others.—See XVI.

PATENTS.

Treatment of the higher phenols for germicidal purposes. V. LEONARD (B.P. 299,522, 29.7.27).—A germicidal solution contains 1 pt. of hexaresorcinol in a mixture of 300 pts. of glycerol and 700 pts. of water. A. COLES.

Chemical purification of waters with a high content of lime and magnesia. E. J. H. BUNCE (F.P. 624,101, 5.11.26. Tunis, 21.6.26).—The water is treated with the calculated quantity of a mixture of disodium hydrogen phosphate and sodium hydrogen carbonate. A. R. POWELL.

Water softening. P. E. LEISS (U.S.P. 1,689,036, 23.10.28. Appl., 1.11.23).—A mixture of barium carbonate and calcium hydroxide in water is added to the water to be softened. C. A. KING.

Conditioning or treating water in boilers etc. to prevent incrustation and corrosion. J. GORDON (B.P. 299,073—4, 17.5.27).—(A) Sodium carbonate or bicarbonate, or, preferably when the gauge pressure exceeds 210 lb./in.², (B) sodium phosphate, fluoride, or arsenate, is added to the water in the boilers to precipitate calcium, magnesium, etc. as a sludge, and further quantities are added as evaporation proceeds. Portions of the water are removed continuously from the boiler and returned again after filtration. L. A. COLES.

Means for preventing boiler-scale. J. PIERON (F.P. 623,440, 3.2.26).—A solution of zinc chloride or sulphate and extract of logwood is claimed. A. R. POWELL.

Means for preventing boiler-scale. W. LAZARUS (F.P. 623,808, 29.10.26. Ger., 20.11.25).—A paste of soot and water is claimed. A. R. POWELL.

Dealing with floating sludge in digestion chambers for sewage treatment. K. IMHOFF and F. FRIES (U.S.P. 1,690,682, 6.11.28. Appl., 25.8.24. Ger., 7.5.24).—See B.P. 230,733; B., 1925, 421.

Means [pads] for disinfecting and/or purifying the atmosphere. L. SCHOENFELD (B.P. 299,380, 20.7.27).

Water-softening apparatus. H. BARON. From W. NEUMANN (B.P. 298,997, 19.7.27).